DEPARTMENT OF THE AIR FORCE
377TH AIR BASE WING (AFGSC)

JUL 29 2016

Colonel Eric H. Froehlich
377 ABW/CC
2000 Wyoming Blvd SE
Kirtland AFB NM 87117-5000

Mr. John Kieling, Bureau Chief
Hazardous Waste Bureau (HWB)
New Mexico Environment Department (NMED)
2905 Rodeo Park Drive East, Building 1
Santa Fe NM 87505-6303

Dear Mr. Kieling

Please find attached the revised *Work Plan for Soil Vapor Monitoring and Drinking Water Monitoring, August 2016, Solid Waste Management Unit ST-106/SS-111,* Kirtland Air Force Base (AFB), New Mexico. This work plan has been revised and updated in response to NMED's letter dated June 30, 2016.

If you have any question or concerns, please contact Mr. Wayne Bitner at (505) 853-3484 or at ludie.bitner@us.af.mil.

Sincerely

ERIC H. FROEHLICH, Colonel, USAF
Commander

Attachment:
Work Plan for Soil Vapor Monitoring and Drinking Water Monitoring, BFF, August 2016; (2) Hard copies & (2) CDs

cc:
NMED-EHD (Roberts, McQuillan), letter/CD
NMED-HWB (Agnew), letter/CD
EPA Region 6 (King, Ellinger), letter/CD
SAF-IEE (Lynnes), letter only
AFCEC/CZ (Bodour, Bitner, Clark), letter only
USACE-ABQ District Office (Simpler, Phaneuf, Dreeland, Sanchez, Salazar), letter only
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WORK PLAN FOR
SOIL VAPOR MONITORING AND DRINKING WATER MONITORING
SOLID WASTE MANAGEMENT UNIT
ST-106/SS-111

August 2016

377 MSG/CEANR
2050 Wyoming Blvd. SE
Kirtland AFB, New Mexico 87117-5270

KIRTLAND AIR FORCE BASE
ALBUQUERQUE, NEW MEXICO

Work Plan for
Soil Vapor Monitoring and
Drinking Water Monitoring
Solid Waste Management Unit ST-106/SS-111

August 2016

Prepared for

U.S. Army Corps of Engineers
Albuquerque District
Albuquerque, New Mexico 87109

USACE Contract No. W912PP-16-C-0002

Prepared by

Sundance Consulting, Inc.
8210 Louisiana Blvd, Suite C
Albuquerque, NM 87113

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REPORT DOCUMENTATION PAGE

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R. Hobbs, P.G.

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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Albuquerque, NM 87113

8. PERFORMING ORGANIZATION REPORT NUMBER

KAFB-16-002

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

USACE Albuquerque District

4101 Jefferson Plaza NE Albuquerque, NM 87109

Project Manager: Trent Simpler, P.E.

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

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12b. DISTRIBUTION CODE

13. This Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP) has been prepared by Sundance Consulting, Inc. as part of the ongoing monitoring effort at Solid Waste Management Unit (SWMU) ST-106/SS-111 at the Kirtland Air Force Base (KAFB) Bulk Fuels Facility. The WP demonstrates the U.S. Air Force's commitment to continuing with the treatment of the fuel contamination resulting from past practices and events at SWMU ST-106/SS-111. This WP outlines activities to be performed in support of continued monitoring of drinking water and the nature and extent of soil vapor contamination at SWMU ST-106/SS-111, and in conjunction with the Quality Assurance Project Plan, will become the procedural guidance document for these activities. This document meets the most recent requirements of the Department of Defense (DoD) regarding planning documents for DoD facilities. The WP was written in accordance with KAFB's Resource Conservation and Recovery Act Permit Number NM9570024423.

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40 CFR 270.11
DOCUMENT CERTIFICATION
AUGUST 2016

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision according to a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowing violations.

ERIC H. FROEHLICH, Colonel, USAF
Commander, 377th Air Base Wing

This document has been approved for public release.

Adhana Zuch

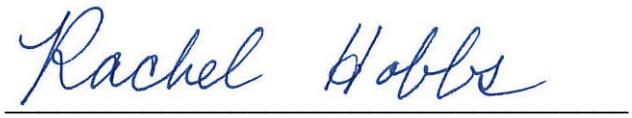
KIRTLAND AIR FORCE BASE
377th Air Base Wing Public Affairs

PREFACE

This Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP) was prepared by Sundance, Consulting, Inc. (Sundance) for the U.S. Army Corps of Engineers (USACE) under contract W912PP-16-C-0002. It pertains to the Kirtland Air Force Base (KAFB) Bulk Fuels Facility site at Solid Waste Management Unit ST-106/SS-111, located in Albuquerque, New Mexico. This WP was prepared in accordance with the permit issued to KAFB under the Resource Conservation and Recovery Act and applicable federal, state, and local laws and regulations.

This WP presents and describes all activities associated with the quarterly sampling of 284 soil vapor monitoring points and the monthly sampling of four drinking water production wells.

This WP is prepared for work to be performed between 20 January 2016 and 20 January 2018. Ms. Amy Sanchez is the Contracting Officer's Representative for the USACE Albuquerque District, and Mr. Trent Simpler, Professional Engineer, is the Project Manager. Mr. Wayne Bitner, Jr. is the KAFB Restoration Section Chief. This plan was prepared by Rachel Hobbs, P.G, the Sundance Project Manager.



Rachel Hobbs, P.G.
Sundance Consulting, Inc.
Project Manager

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ACRONYMS AND ABBREVIATIONS

%	percent
^{o}C	degrees Celsius
AFB	Air Force Base
AFCEC	Air Force Civil Engineering Center
Air Force	U.S. Air Force
ALS	ALS Environmental Laboratories
APH	air-phase petroleum hydrocarbons
APP	Accident Prevention Plan
BFF	Bulk Fuels Facility
BTEX	benzene, toluene, ethylbenzene, and xylenes
CFR	Code of Federal Regulations
CO_2	carbon dioxide
COA	City of Albuquerque
CY	calendar year
DO	dissolved oxygen
DoD	U.S. Department of Defense
DTIC	Defense Technical Information Center
EDB	ethylene dibromide
e.g.	example given
EPA	U.S. Environmental Protection Agency
ERP	Environmental Restoration Program
ERPIMS	Environmental Resource Program Information Management System
HC	hydrocarbons
Horiba	Horiba MEXA 584L auto emissions analyzer
IDW	investigation-derived waste
in Hg	inches of mercury
in WC	inches of water column
KAFB	Kirtland Air Force Base
LDC	Laboratory Data Consultants, Inc.
NMED	New Mexico Environment Department
O_2	oxygen
ORP	oxidation reduction potential
P.E.	Professional Engineer
P.G.	Professional Geologist
PID	photoionization detector
PPE	personal protective equipment
ppmv	parts per million by volume

QAPjP	Quality Assurance Project Plan
QC	quality control
QMR	Quarterly Monitoring and Site Investigation Report
RCRA	Resource Conservation and Recovery Act
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
Sundance	Sundance Consulting, Inc.
SVE	soil vapor extraction
SVEW	soil vapor extraction well
SVM	soil vapor monitoring
SVMP	soil vapor monitoring point
SWMU	Solid Waste Management Unit
SWMW	soil vapor monitoring well
U.S.	United States
USACE	U.S. Army Corps of Engineers
VA	Veteran's Administration
VOC	volatile organic compound
WP	Work Plan
YSI	Yellow Springs Instruments

EXECUTIVE SUMMARY

This Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP) has been prepared by Sundance Consulting, Inc. as part of the ongoing monitoring effort at Solid Waste Management Unit (SWMU) ST-106/SS-111 at the Kirtland Air Force Base (KAFB) Bulk Fuels Facility. The WP demonstrates the U.S. Air Force's commitment to continuing with the treatment of the fuel contamination resulting from past practices and events at SWMU ST-106/SS-111. This WP outlines activities to be performed in support of continued monitoring of drinking water and the nature and extent of soil vapor contamination at SWMU ST-106/SS-111, and in conjunction with the Quality Assurance Project Plan, will become the procedural guidance document for these activities. These documents meet the most recent requirements of the Department of Defense (DoD) regarding planning documents for DoD facilities. The WP was written in accordance with KAFB's Resource Conservation and Recovery Act Permit Number NM9570024423.

The objective of the WP is to detail the quarterly sampling and analysis of the existing soil vapor monitoring network, and monthly drinking water well sampling and analysis activities to be implemented. The work to be completed is presented under each of the tasks listed below:

- Perform quarterly sampling and reporting at 284 soil vapor monitoring points for 8 quarters beginning in first quarter calendar year (CY) 2016.
- Perform yearly maintenance of the soil vapor monitoring network.
- Abandon and install soil vapor monitoring locations.
- Perform monthly sampling and reporting of four drinking water production wells beginning in February 2016 through the end of CY 2017.

1 INTRODUCTION

This Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP) was prepared by Sundance, Consulting, Inc. (Sundance) for the U.S. Army Corps of Engineers (USACE) under contract W912PP-16-C-0002. The WP pertains to sampling activities at the Kirtland Air Force Base (KAFB) Bulk Fuels Facility (BFF), Solid Waste Management Unit (SWMU) ST-106/SS-111 (example given [e.g.] BFF site). Environmental restoration efforts at the BFF site are being conducted under requirements set forth in the Resource Conservation and Recovery Act (RCRA) Permit No. NM9570024423 with the New Mexico Environment Department (NMED) serving as the lead regulatory agency (NMED, 2010).

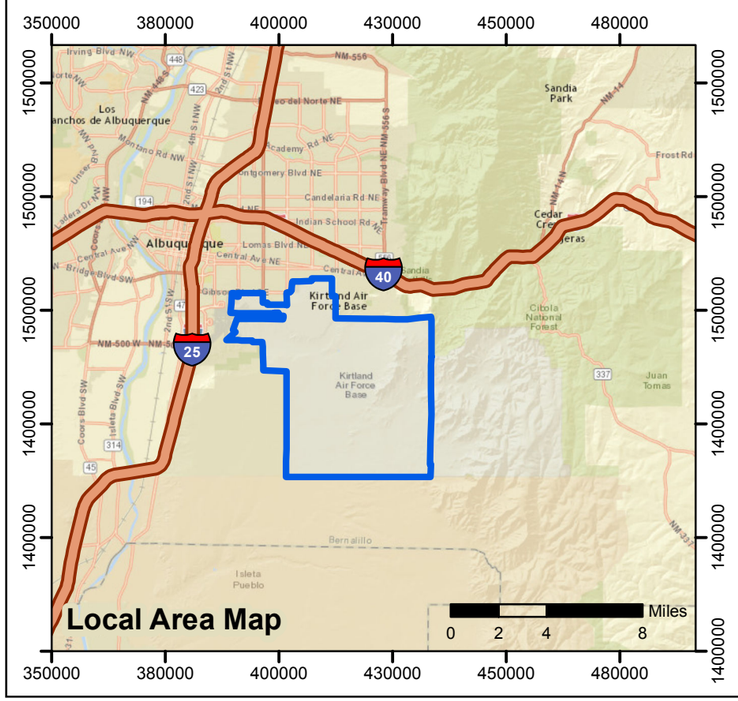
This WP addresses activities that are continuing the implementation of the RCRA process for this site, including the continued monitoring of the soil vapor plume, and monitoring of drinking water production wells. This WP will become the procedural guidance document for these activities to be performed as part of the ongoing investigation and will meet the most recent requirements of the Department of Defense (DoD) regarding planning documents for DoD facilities.

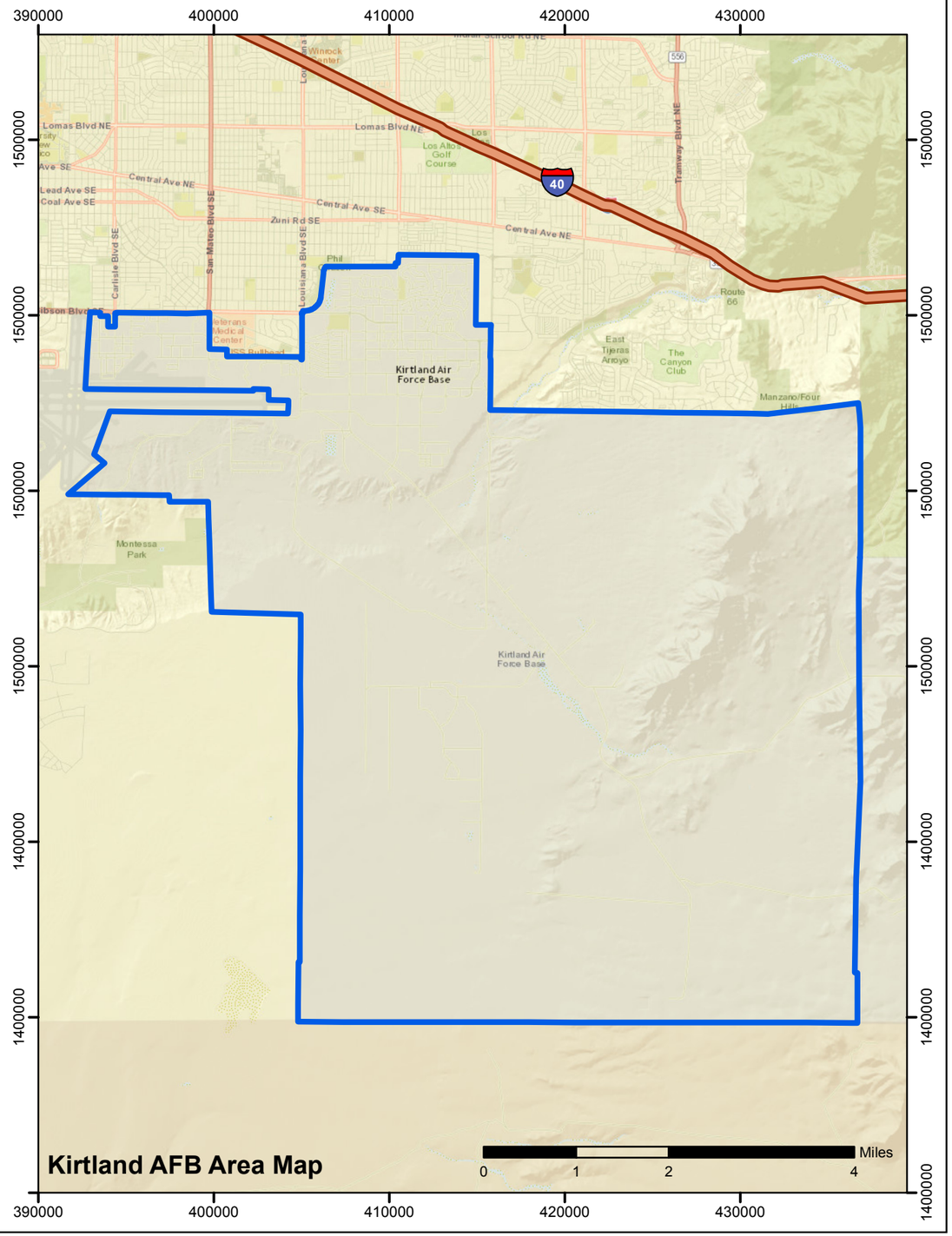
Requirements for the protection of health and safety on the job sites are addressed in the companion *Quarterly Soil Vapor Sampling and Monthly Drinking Water Sampling Accident Prevention Plan* (APP) (Sundance 2016a). The APP also incorporates the Site Safety and Health Plan (SSHP).

1.1 Overview and Scope of Activities

The BFF site is located in Albuquerque, New Mexico (Figure 1-1). Field activities presented in this WP include quarterly soil vapor sampling and analysis of 284 monitoring points installed as part of the investigation at SWMU ST-106/SS-111. In addition, monthly sampling and analysis of four drinking water production wells will be performed. Analytical results from soil vapor and drinking water samples will be reported in the Quarterly Monitoring and Site Investigation Reports (QMRs). Analytical results from the drinking water production well samples will be reported in the QMRs. This WP for quarterly soil vapor sampling and monthly drinking water sampling includes all of the elements of a Sampling and Analysis Plan/Field Sampling Plan and covers all the project tasks associated with:

- Sample soil vapor monitoring network quarterly, beginning in first quarter calendar year (CY) 2016 for eight (8) quarters.
- Sample drinking water production wells monthly, beginning in February 2016 through the end of CY 2017.
- Perform annual maintenance of the soil vapor monitoring network.
- Abandon and install soil vapor monitoring locations.
- Analyze soil vapor and drinking water production well samples, and report results quarterly.





Legend

- Interstate
- US Highway
- State/County Highway
- States
- Kirtland Air Force Base Installation Area

N

Credits: Esri, HERE, DeLorme, USGS, Intermap

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

Figure 1-1

Site Location Map
Work Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016

1.2 Report Organization

The BFF Soil Vapor Monitoring and Drinking Water Monitoring WP is divided into the following sections:

- Section 1—Presents an introduction to the plan, an overview of the project, and the scope of activities and organization of the WP.
- Section 2—Presents the BFF site description and operational history.
- Section 3—Project tasks are summarized with sufficient detail on how they will be accomplished.
- Section 4—Presents the project schedule.
- Section 5—Provides information on the organizational plan for the execution of work.
- Section 6—Refers to the data management requirements.
- Section 7—Presents information on the management and disposal of the waste generated during this project.
- Section 8—Presents references cited for this WP.

Associated appendices are provided at the end of this WP as follows:

- Appendix A: Field Forms
  -Field Activity Log
  -Well Integrity Checklist
  -Horiba Calibration Form
  -Leak Test Log
  -Example Soil Vapor Purge Log
  -Example Soil Vapor Sample Collection Log
  -Example Soil Vapor Chain of Custody
  -Example Water Sample Collection Log
  -Example Water Chain of Custody
- Appendix B: Project Schedule
- Appendix C: Quality Assurance Project Plan (QAPjP)

2 BACKGROUND INFORMATION

2.1 Site Description

KAFB is located in Bernalillo County, in central New Mexico, southeast of and adjacent to the City of Albuquerque (COA) and the Albuquerque International Sunport (Figure 1-1). The approximate area of the base is 52,287 acres. The BFF site is located in the northwestern portion of KAFB.

2.2 Site History

The BFF and associated infrastructure operated from 1953 through 1999. During this time, the fueling area was separated into a tank holding area where bulk shipments of fuel were received and a fuel off-loading area where individual fuel railcars or trucks were emptied. KAFB stopped using the underground piping at the facility in 1999 due to discovery of leaks in buried fuel transfer piping.

Even though a fuel leak was identified by KAFB in 1999, the exact history of the leaks or releases is unknown. Releases most likely occurred when fuel was transferred from railcars and trucks to the pump house. Initially, it was thought that the leak only affected surface soil around the identified source area; however, during site characterization activities KAFB learned the leaked fuel had reached the groundwater table and that dissolved-phase fuel contamination migrated northeast and north of KAFB.

2.3 Ongoing Monitoring

2.3.1 Ongoing Soil Vapor Monitoring

Quarterly soil vapor monitoring has been ongoing under the *Vadose Zone Investigation Work Plan* (USACE, 2011) as part of the ST-106/SS-111 investigation to monitor the nature and extent of soil vapor contamination in the vadose zone. A total of 56 soil vapor monitoring locations have been installed during the investigation (Figure 2-1). Each location is comprised of one or more soil vapor monitoring points (SVMPs), for a total of 284 monitoring points. Table 2-1 lists each soil vapor monitoring location, its associated SVMPs, and their associated easting and northing coordinates. The 56 soil vapor monitoring locations include:

- One soil vapor monitoring location contains four SVMPs co-located in the same vault as a groundwater monitoring well in Bullhead Park (KAFB-106028-510; See location number one in Table 2-1 below).

- Thirty-five locations installed in 2010 and 2011, each with six SVMPs, are located throughout the BFF, on base property north of the BFF, on COA property in Bullhead Park and its open space area, and on Veteran's Administration (VA) property. These locations are named using the convention KAFB-106XXX, to signify that they were installed as part of the investigation at SWMU ST-106/SS-111. Numbering at these 35 soil vapor monitoring locations range from KAFB-106108 through KAFB-106142 (See locations two through 36 in Table 2-1 below). Individual SVMPs at each location are further identified using the bottom of the screen depth of each point (e.g. KAFB-106108-050).

- Twenty locations installed inside the BFF referred to with the prefix "soil vapor extraction well (SVEW)-XX" or "soil vapor monitoring well (SVMW)-XX (See locations 37 through 56 in table 2-1 below)."

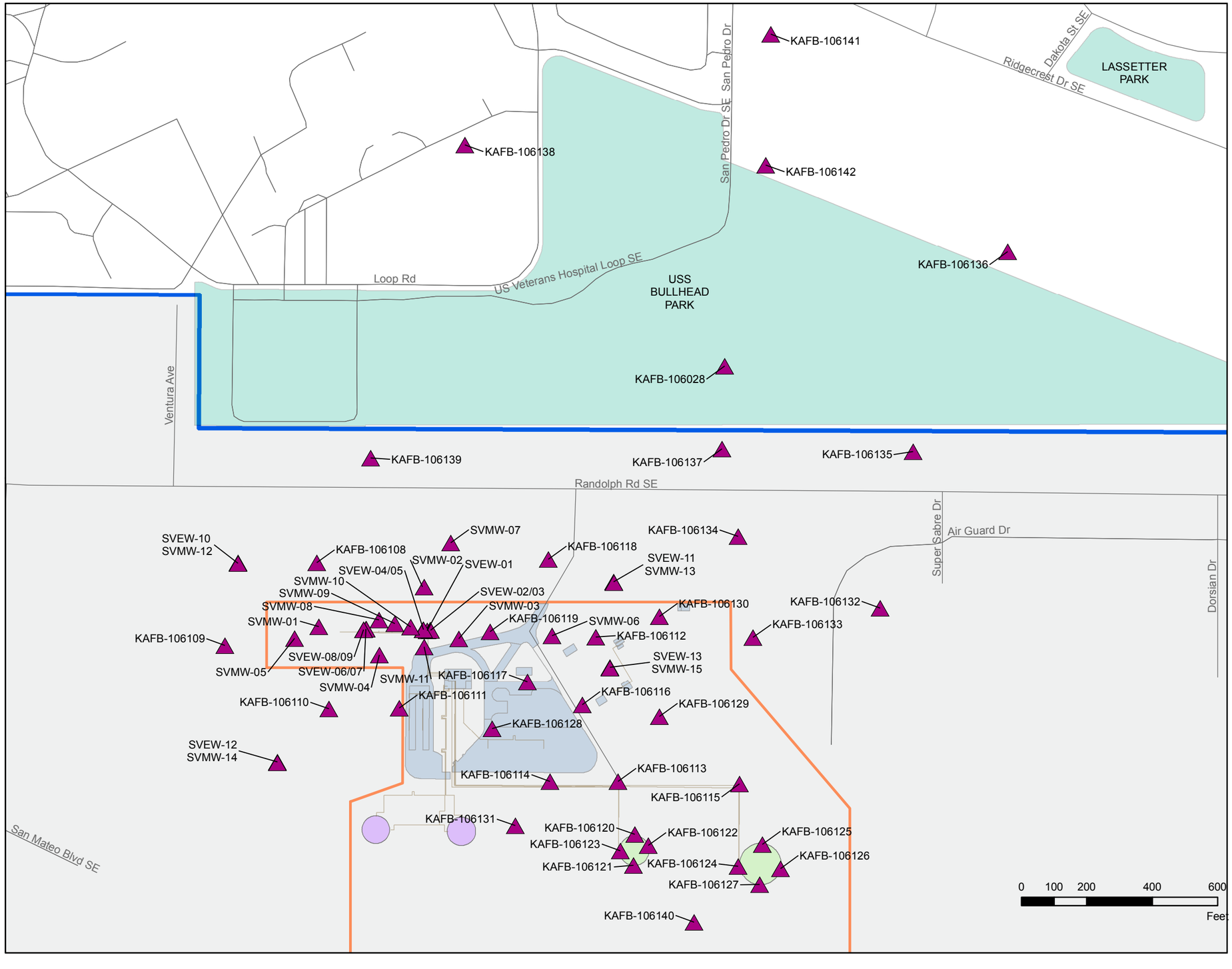
    - Four of these 20 locations have both SVMW and SVEW type SVMPs at a single location. [For example, SVMW-13 (comprised of four SVMPs screened between 150

and 450-feet deep) and SVEW-11 (with one SVMP) are located together in a single well vault.

Table 2-1 lists each soil vapor monitoring well location and its associated SVMPs and coordinates. In first quarter CY 2015, all SVMPs were capped and sealed to minimize barometric-pumping interferences on soil vapor sampling and analyses. Sealing the SVMPs was performed by securing an air-tight cap onto each point/well head and adding a pneumatic quick connect fitting to each monitoring point that serves as a sampling port connection for ease of access and to ensure that an air-tight seal is maintained.

2.3.2 Ongoing Drinking Water Production Well Monitoring

Four drinking water production wells have been sampled monthly as part of the ST-106/SS-111 investigation to confirm that they have not been impacted by groundwater contaminants. These wells include ST106-VA2 on VA hospital property, and KAFB-3, KAFB-15 and KAFB-16 on KAFB property (Figure 2-2). Table 2-2 lists the coordinates of each drinking water well.



Legend

- Kirtland Air Force Base Installation Area
- City of Albuquerque Parks
- Roads
- Bulk Fuels Facility Area
- Soil Vapor Monitoring Location
- Bulk Fuels Facility Infrastructure
- Current Fuel Storage Tanks
- Former Fuel Storage Tanks
- Fuel Transfer Lines

N

Credits: City of Albuquerque

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

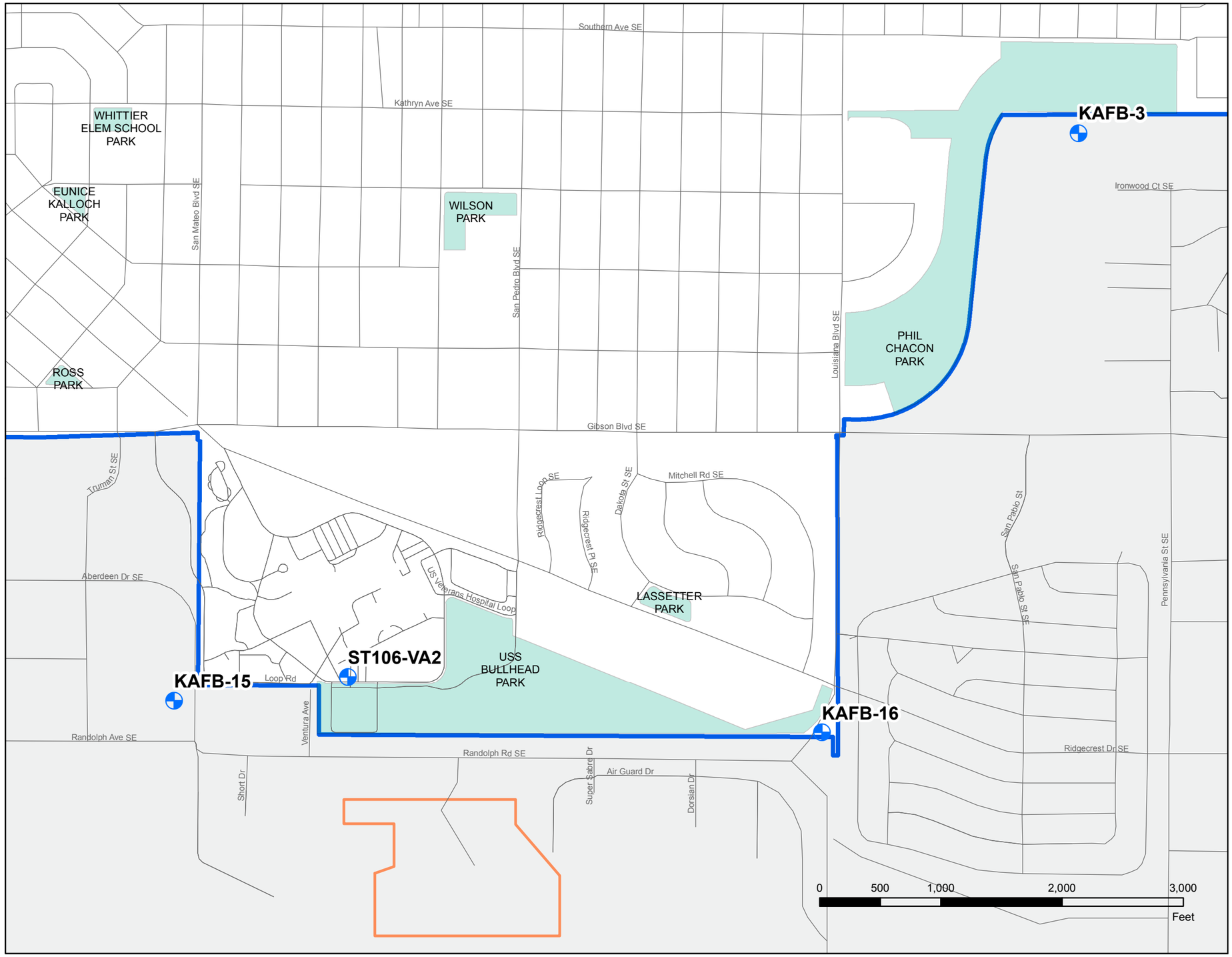
Figure 2-1

Soil Vapor Monitoring Locations
Work Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016



Legend

- Kirtland Air Force Base Installation Area
- City of Albuquerque Parks
- Roads
- Bulk Fuels Facility Area
- Drinking Water Supply Well

N

Credits: City of Albuquerque

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

Figure 2-2

Drinking Water Supply Wells
Work Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
1	KAFB-106028	KAFB-106028-150	401910.441776	1474285.009804
		KAFB-106028-250	401910.491777	1474285.239798
		KAFB-106028-350	401910.751783	1474285.259797
		KAFB-106028-450	401910.851786	1474285.409793
2	KAFB-106108	KAFB-106108-025	400664.903033	1473684.446268
		KAFB-106108-050	400664.773030	1473684.206274
		KAFB-106108-150	400664.993035	1473684.016279
		KAFB-106108-250	400665.243040	1473684.156276
		KAFB-106108-350	400665.173039	1473684.456267
		KAFB-106108-450	400665.023035	1473684.846257
3	KAFB-106109	KAFB-106109-025	400384.736587	1473432.363139
		KAFB-106109-050	400384.916591	1473432.573133
		KAFB-106109-150	400384.736587	1473432.853126
		KAFB-106109-250	400384.456580	1473432.763128
		KAFB-106109-350	400384.446580	1473432.443137
		KAFB-106109-450	400384.676586	1473432.693130
4	KAFB-106110	KAFB-106110-025	400702.613980	1473238.228342
		KAFB-106110-050	400702.373975	1473238.008348
		KAFB-106110-150	400702.523978	1473237.768354
		KAFB-106110-250	400702.783984	1473237.818353
		KAFB-106110-350	400702.833985	1473238.098345
		KAFB-106110-450	400702.033967	1473237.748355
5	KAFB-106111	KAFB-106111-025	400916.998944	1473240.918233
		KAFB-106111-050	400917.178948	1473240.698239
		KAFB-106111-150	400917.438954	1473240.808236
		KAFB-106111-250	400917.418954	1473241.068229
		KAFB-106111-350	400917.118947	1473241.148227
		KAFB-106111-450	400917.038945	1473240.748238
6	KAFB-106112	KAFB-106112-025	401517.592816	1473457.832261
		KAFB-106112-050	401517.752819	1473457.592267
		KAFB-106112-150	401518.032826	1473457.692264
		KAFB-106112-250	401518.062827	1473457.962257
		KAFB-106112-350	401517.772820	1473458.082254
		KAFB-106112-450	401517.052803	1473457.742263
7	KAFB-106113	KAFB-106113-020	401585.424459	1473016.904187
		KAFB-106113-050	401585.284456	1473016.594195
		KAFB-106113-150	401585.534462	1473016.404200
		KAFB-106113-250	401585.844469	1473016.554196
		KAFB-106113-350	401585.754467	1473016.824189
		KAFB-106113-450	401585.554462	1473016.764191

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
8	KAFB-106114	KAFB-106114-025	401377.539646	1473017.104216
		KAFB-106114-050	401377.289640	1473016.884222
		KAFB-106114-150	401377.419643	1473016.594230
		KAFB-106114-250	401377.729650	1473016.614229
		KAFB-106114-350	401377.759651	1473016.924221
		KAFB-106114-450	401377.589647	1473017.374209
9	KAFB-106115	KAFB-106115-025	401956.373050	1473009.614323
		KAFB-106115-050	401956.193046	1473009.374329
		KAFB-106115-150	401955.923040	1473009.414328
		KAFB-106115-250	401955.893039	1473009.694321
		KAFB-106115-350	401956.163045	1473009.754319
		KAFB-106115-450	401956.063043	1473009.754319
10	KAFB-106116	KAFB-106116-025	401475.631878	1473251.167863
		KAFB-106116-050	401475.951886	1473251.187862
		KAFB-106116-150	401475.521876	1473251.417856
		KAFB-106116-250	401475.741881	1473251.627850
		KAFB-106116-350	401476.021887	1473251.467854
		KAFB-106116-450	401475.051865	1473250.747874
11	KAFB-106117	KAFB-106117-025	401308.888006	1473321.085997
		KAFB-106117-050	401309.028009	1473321.305991
		KAFB-106117-150	401308.838004	1473321.555985
		KAFB-106117-250	401308.567998	1473321.445988
		KAFB-106117-350	401308.577998	1473321.165995
		KAFB-106117-450	401308.047986	1473320.746007
12	KAFB-106118	KAFB-106118-025	401372.389414	1473695.475851
		KAFB-106118-050	401372.479416	1473695.765843
		KAFB-106118-160	401372.589419	1473695.285856
		KAFB-106118-265	401372.809424	1473695.735844
		KAFB-106118-350	401372.879425	1473695.385854
		KAFB-106118-450	401372.049406	1473695.735844
13	KAFB-106119	KAFB-106119-025	401194.105322	1473474.311868
		KAFB-106119-050	401194.275326	1473474.061875
		KAFB-106119-150	401194.545332	1473474.151873
		KAFB-106119-250	401194.585333	1473474.421865
		KAFB-106119-350	401194.315327	1473474.531862
		KAFB-106119-450	401194.045321	1473474.741857
14	KAFB-106120	KAFB-106120-025	401636.035658	1472855.058560
		KAFB-106120-050	401636.135660	1472855.248555
		KAFB-106120-150	401636.015658	1472855.498549
		KAFB-106120-250	401635.685650	1472855.268555
		KAFB-106120-350	401635.855654	1472855.038561
		KAFB-106120-450	401635.915655	1472855.388552

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
15	KAFB-106121	KAFB-106121-025	401632.585594	1472760.611118
		KAFB-106121-050	401632.835600	1472760.431123
		KAFB-106121-145	401632.305587	1472760.381125
		KAFB-106121-250	401632.425590	1472760.121132
		KAFB-106121-350	401632.805599	1472760.141131
		KAFB-106121-450	401632.535593	1472760.431123
16	KAFB-106122	KAFB-106122-025	401677.386621	1472821.089473
		KAFB-106122-050	401677.326620	1472821.389465
		KAFB-106122-150	401677.046613	1472821.449464
		KAFB-106122-250	401676.936611	1472821.159472
		KAFB-106122-350	401677.156616	1472820.949477
		KAFB-106122-450	401677.146616	1472821.289468
17	KAFB-106123	KAFB-106123-025	401591.874644	1472805.169919
		KAFB-106123-050	401591.754641	1472805.439911
		KAFB-106123-150	401591.394633	1472805.419912
		KAFB-106123-250	401591.334631	1472805.109920
		KAFB-106123-350	401591.614638	1472804.929925
		KAFB-106123-450	401591.564637	1472805.319915
18	KAFB-106124	KAFB-106124-025	401951.482979	1472757.341154
		KAFB-106124-050	401951.172971	1472757.361154
		KAFB-106124-150	401951.042968	1472757.101161
		KAFB-106124-250	401951.282974	1472756.901166
		KAFB-106124-350	401951.522980	1472757.031163
		KAFB-106124-450	401951.302974	1472757.261156
19	KAFB-106125	KAFB-106125-025	402026.084695	1472824.439325
		KAFB-106125-050	402025.794688	1472824.509323
		KAFB-106125-150	402025.624684	1472824.279330
		KAFB-106125-250	402025.824689	1472824.009337
		KAFB-106125-350	402026.094695	1472824.099334
		KAFB-106125-450	402025.864690	1472824.369327
20	KAFB-106126	KAFB-106126-025	402081.045980	1472750.941306
		KAFB-106126-050	402081.015979	1472751.231298
		KAFB-106126-150	402080.725972	1472751.321296
		KAFB-106126-250	402080.555968	1472751.081302
		KAFB-106126-350	402080.735973	1472750.781310
		KAFB-106126-450	402080.795974	1472751.161300
21	KAFB-106127	KAFB-106127-025	402018.094530	1472701.832646
		KAFB-106127-050	402017.834524	1472701.972642
		KAFB-106127-150	402017.604519	1472701.792647
		KAFB-106127-250	402017.754522	1472701.472656
		KAFB-106127-350	402018.054529	1472701.512655
		KAFB-106127-450	402017.914526	1472701.832646

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
22	KAFB-106128	KAFB-106128-025	401200.655523	1473177.999889
		KAFB-106128-050	401200.545521	1473177.729897
		KAFB-106128-150	401200.765526	1473177.529902
		KAFB-106128-250	401201.025532	1473177.669898
		KAFB-106128-350	401200.965530	1473177.999889
		KAFB-106128-450	401200.045509	1473177.749896
23	KAFB-106129	KAFB-106129-025	401711.597348	1473215.518789
		KAFB-106129-050	401711.877355	1473215.378792
		KAFB-106129-150	401712.097360	1473215.618786
		KAFB-106129-250	401711.977357	1473215.878779
		KAFB-106129-350	401711.687350	1473215.848780
		KAFB-106129-450	401711.817353	1473216.128772
24	KAFB-106130	KAFB-106130-025	401711.947306	1473520.410534
		KAFB-106130-050	401712.157311	1473520.280538
		KAFB-106130-150	401712.427317	1473520.380535
		KAFB-106130-250	401712.377316	1473520.660528
		KAFB-106130-350	401712.107310	1473520.740525
		KAFB-106130-450	401712.057308	1473520.740525
25	KAFB-106131	KAFB-106131-025	401271.447212	1472881.677900
		KAFB-106131-055	401271.667217	1472881.497905
		KAFB-106131-150	401271.567214	1472881.987892
		KAFB-106131-245	401271.937223	1472881.657901
		KAFB-106131-350	401271.837220	1472881.947893
		KAFB-106131-450	401271.047202	1472881.757898
26	KAFB-106132	KAFB-106132-025	402385.372896	1473546.049729
		KAFB-106132-050	402384.882885	1473546.139727
		KAFB-106132-175	402385.202892	1473545.799736
		KAFB-106132-250	402384.892885	1473545.829735
		KAFB-106132-350	402385.152891	1473546.259724
		KAFB-106132-450	402385.072889	1473545.739738
27	KAFB-106133	KAFB-106133-025	401997.193922	1473456.332222
		KAFB-106133-050	401997.113920	1473456.612214
		KAFB-106133-170	401997.353925	1473456.832208
		KAFB-106133-250	401997.593931	1473456.672213
		KAFB-106133-350	401997.543930	1473456.362221
		KAFB-106133-450	401997.063919	1473456.742211
28	KAFB-106134	KAFB-106134-025	401952.322832	1473764.643883
		KAFB-106134-050	401952.042825	1473764.673882
		KAFB-106134-170	401952.432834	1473764.923875
		KAFB-106134-250	401952.232830	1473765.113870
		KAFB-106134-350	401951.932823	1473764.953875
		KAFB-106134-450	401952.282831	1473765.433862

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
29	KAFB-106135	KAFB-106135-025	402486.125151	1474023.666783
		KAFB-106135-050	402486.335156	1474023.456789
		KAFB-106135-150	402486.195152	1474023.146797
		KAFB-106135-250	402485.865145	1474023.256795
		KAFB-106135-350	402485.875145	1474023.556786
		KAFB-106135-450	402486.055149	1474023.526787
30	KAFB-106136	KAFB-106136-025	402775.081742	1474634.810194
		KAFB-106136-050	402775.081742	1474634.910191
		KAFB-106136-150	402775.081742	1474635.010188
		KAFB-106136-250	402775.081742	1474635.110186
		KAFB-106136-350	402775.081742	1474635.210183
		KAFB-106136-450	402775.081742	1474635.310180
31	KAFB-106137	KAFB-106137-025	401902.701639	1474032.086651
		KAFB-106137-050	401902.451633	1474032.276646
		KAFB-106137-150	401902.181627	1474032.086652
		KAFB-106137-250	401902.311630	1474031.816659
		KAFB-106137-350	401902.661638	1474031.816659
		KAFB-106137-450	401902.521634	1474034.116597
32	KAFB-106138	KAFB-106138-025	401117.783308	1474960.381655
		KAFB-106138-050	401117.443300	1474960.271658
		KAFB-106138-150	401117.413299	1474959.981666
		KAFB-106138-250	401117.693306	1474959.871669
		KAFB-106138-350	401117.913311	1474960.121662
		KAFB-106138-450	401117.613304	1474960.211660
33	KAFB-106139	KAFB-106139-025	400829.366788	1474004.577574
		KAFB-106139-050	400829.566792	1474004.137586
		KAFB-106139-150	400829.296786	1474004.027589
		KAFB-106139-250	400829.156783	1474004.327581
		KAFB-106139-350	400829.636794	1474004.477577
		KAFB-106139-450	400829.556792	1474004.807568
34	KAFB-106140	KAFB-106140-025	401817.689909	1472587.305780
		KAFB-106140-050	401818.019916	1472587.175784
		KAFB-106140-150	401817.719909	1472587.635771
		KAFB-106140-250	401818.019916	1472587.695770
		KAFB-106140-350	401818.249922	1472587.445776
		KAFB-106140-450	401817.949915	1472587.515774
35	KAFB-106141	KAFB-106141-025	402051.934886	1475298.122360
		KAFB-106141-050	402052.064889	1475298.382353
		KAFB-106141-170	402051.904885	1475298.602347
		KAFB-106141-250	402051.614878	1475298.502350
		KAFB-106141-350	402051.644879	1475298.222357
		KAFB-106141-450	402051.844884	1475298.452351

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
36	KAFB-106142	KAFB-106142-030	402036.744600	1474898.733172
		KAFB-106142-050	402036.494594	1474898.863168
		KAFB-106142-170	402036.284589	1474898.643174
		KAFB-106142-250	402036.434593	1474898.363182
		KAFB-106142-350	402036.734600	1474898.453179
		KAFB-106142-450	402036.554596	1474898.703173
37	SVEW-01	SVEW-01	401002.570886	1473477.521813
38	SVEW-02/03	SVEW-02/03-060	401012.021105	1473479.611755
		SVEW-02/03-160	401012.091107	1473478.491785
39	SVEW-04/05	SVEW-04/05-313	400990.430605	1473479.771754
		SVEW-04/05-460	400991.320625	1473479.841752
40	SVEW-06/07	SVEW-06/07-060	400815.436552	1473481.961724
		SVEW-06/07-160	400816.096568	1473481.921725
41	SVEW-08/09	SVEW-08/09-260	400807.566370	1473480.611762
		SVEW-08/09-460	400807.936379	1473480.271771
42	SVMW-01	SVMW-01-050	400671.123209	1473489.171553
		SVMW-01-100	400671.443217	1473489.531543
		SVMW-01-250	400671.423216	1473489.171553
		SVMW-01-300	400671.163210	1473489.621541
43	SVMW-02	SVMW-02-050	400993.070644	1473610.618212
		SVMW-02-100	400993.170647	1473610.478215
		SVMW-02-150	400993.220648	1473610.748208
44	SVMW-03	SVMW-03-050	401099.413133	1473452.122485
		SVMW-03-100	401099.553136	1473452.472475
		SVMW-03-250	401099.743141	1473452.272481
		SVMW-03-300	401099.633138	1473451.992488
45	SVMW-04	SVMW-04-050	400855.887502	1473403.243849
		SVMW-04-100	400855.527494	1473403.383845
		SVMW-04-250	400855.527494	1473403.113852
		SVMW-04-300	400855.697498	1473402.813860
46	SVMW-05	SVMW-05-050	400597.061500	1473453.092542
		SVMW-05-100	400596.771493	1473453.362535
		SVMW-05-230	400597.201503	1473453.422533
		SVMW-05-290	400596.971498	1473453.502531
47	SVMW-06	SVMW-06-050	401383.549711	1473462.182165
		SVMW-06-100	401384.009722	1473462.052169
		SVMW-06-252	401383.769716	1473462.402159
		SVMW-06-302	401383.689714	1473461.872174
48	SVMW-07	SVMW-07-050	401074.372504	1473745.824538
		SVMW-07-100	401074.072497	1473745.944535
		SVMW-07-150	401074.272502	1473746.164529

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
49	SVMW-08	SVMW-08-050	400855.727481	1473509.800964
		SVMW-08-100	400855.777482	1473510.000958
		SVMW-08-250	400855.807482	1473509.770965
50	SVMW-09	SVMW-09-050	400903.768595	1473499.001248
		SVMW-09-100	400903.668593	1473498.981249
		SVMW-09-250	400903.458588	1473499.251241
		SVMW-09-266	400903.188581	1473499.171244
51	SVMW-10	SVMW-10-050	400952.179718	1473488.861515
		SVMW-10-100	400952.159717	1473489.341502
		SVMW-10-150	400952.479724	1473489.071509
		SVMW-10-250	400952.069715	1473488.961512
52	SVMW-11	SVMW-11-050	400992.780668	1473428.233149
		SVMW-11-100	400992.940671	1473428.593139
		SVMW-11-250	400992.410659	1473428.593140
		SVMW-11-260	400992.560663	1473428.813134
53	SVMW-12/ SVEW-10	SVMW-12-150	400424.977477	1473683.186342
		SVMW-12-250	400425.217482	1473683.336338
		SVMW-12-350	400424.887475	1473683.766326
		SVMW-12-450	400424.627469	1473683.536333
		SVEW-10	400424.857474	1473685.086291
54	SVMW-13/ SVEW-11	SVMW-13-150	401572.894069	1473624.437741
		SVMW-13-250	401572.554061	1473624.437741
		SVMW-13-350	401572.634063	1473624.217747
		SVMW-13-450	401572.754066	1473623.977754
		SVEW-11	401572.134051	1473626.047698
55	SVMW-14/ SVEW-12	SVMW-14-150	400544.990358	1473074.172810
		SVMW-14-250	400544.730352	1473074.292806
		SVMW-14-350	400544.780353	1473073.942816
		SVMW-14-450	400545.220363	1473073.932816
		SVEW-12	400545.010358	1473076.332751
56	SVMW-15/ SVEW-13	SVMW-15-150	401560.833833	1473362.634831
		SVMW-15-250	401561.343845	1473362.704829
		SVMW-15-350	401561.013837	1473362.434836
		SVMW-15-450	401561.013837	1473362.994821
		SVEW-13	401560.863833	1473364.984767

[1] Well coordinates are provided in New Mexico State Plane (NAD27).

Table 2-2. Drinking Water Supply Well Names and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Well Location Name	Easting	Northing
ST106-VA2	400544.79	1474576.3
KAFB-3	406572.7	1479061.58
KAFB-15	399113.43	1474381.13
KAFB-16	404456.63	1474118.74

3 TASKS AND ACTIVITIES

This section presents the activities that will be performed under this project. Major divisions include mobilization/demobilization, quarterly soil vapor sampling and analysis, and monthly production well sampling and analysis. This section also describes soil vapor monitoring well installation and abandonment, equipment decontamination, use of personal protective equipment (PPE) and photoionization detector (PID), field quality control and sample packaging and shipping.

3.1 Mobilization/Demobilization

A secure, fenced equipment yard has been established for both equipment and materials for sampling activities both on and off KAFB for this project. A portable office trailer with electrical power has been established inside the secured equipment yard proximal to the BFF at KAFB.

3.2 Soil Vapor Sampling

Sundance will perform quarterly sampling of the existing 284 SVMP network described in section 2.3. All field personnel collecting soil vapor samples are required to be trained and fully understand the sampling procedure outlined in this document. Any and all questions will be addressed prior to the start of sampling via a field sampling orientation led by the Sundance Technical Lead. During the first quarter CY 2016 sampling performed by Sundance, the condition of each well port will be examined by the field personnel to confirm the integrity of each fitting and to immediately address and mitigate any problems or replace any defective parts. The well integrity inspection form can be found in Appendix A, Field Forms.

3.2.1 Pre-Sampling Steps

3.2.1.1 Horiba Model MEXA 584L Calibration

During sampling of each soil vapor well, field parameters including total hydrocarbons (HC), oxygen (O_2), and carbon dioxide (CO_2) will be measured using a Horiba Mexa 584L auto emissions analyzer (Horiba).

The Horiba is sold as an engine exhaust monitoring instrument, and the measurement of field parameters during soil vapor monitoring is not the manufacturer's intended purpose. However, while not the intended use of the instrument, the Horiba's sampling ability and the non-dispersive infrared detector and chemical cell detector make it an appropriate instrument for total soil vapor HC, CO_2, and O_2 analyses.

The Horiba manufacturer's calibration procedure, which was developed for engine exhaust monitoring, has been modified to better calibrate the instrument for measuring soil vapor petroleum HC, O_2 and CO_2 concentrations. The modified calibration method includes a more representative calibration gas, more frequent calibration than specified by the manufacturer, frequent calibration checks (HC, O_2 and CO_2) during daily Horiba usage, and real-time data analysis to look for indicators of potential calibration deviations.

At the beginning of every work day, the Horiba will be calibrated for air-phase petroleum HC and CO_2 against a calibration standard of known concentrations in a premixed gas cylinder. The Horiba will also be calibrated for O_2 against atmospheric concentrations. At the middle of each work day (or no more than 5 hours after the start of work), a calibration check will be performed on the Horiba to determine whether the calibration of any of the parameters has drifted since the morning calibration. If the calibration check results are outside of 5% of the calibration gas standards, then the Horiba will be recalibrated prior to additional sampling.

The same calibration gas cylinder will be used to calibrate every Horiba instrument and the same person will complete calibration at the beginning of each day to ensure consistent calibrations. The calibration gas consists of 1,600 parts per million by volume (ppmv) propane, 13.0% (percent) CO_2, and the remaining volume will consist of nitrogen. This calibration gas mixture was selected to accurately calibrate the Horiba for the gases that are present in the vadose zone during soil vapor sampling and respiration/rebound testing, specifically, CO_2 and HC (calibrated as propane).

The initial daily calibration of the instrument will be performed by applying the pre-mixed gas into the calibration port located on top of the instrument. The calibration steps listed in the Horiba manual for applying the calibration gas and the sequence of key strokes listed will be followed to complete calibration. After calibration, HC and CO_2 concentrations should be within 5% of the known calibration gas values.

The Horiba Instruction Manual goes through the calibration of each compound. During the calibration of HC, the Horiba will display three numbers as shown in the Horiba Instruction Manual. The top number is the known value of the calibration gas (propane at 1,600 ppmv). The middle number will be constant for a particular instrument, but will vary from instrument to instrument; this number is the factory calibration setting for that instrument that corresponds to the input value of the calibration gas. The bottom number will change as the calibration gas is applied and read by the instrument. Both the middle and bottom numbers are reporting the gas as hexane rather than propane; hexane is the standard for HC for the Horiba instrument. The conversion between hexane and propane for the Horiba is approximately one half the concentration. Thus, a reading of 800 ppmv reported as hexane by the detector is the equivalent of 1,600 ppmv as propane. Once the gas is applied, the bottom HC number on the display should read within 5% of the middle number. A value of approximately 800 ppmv HC is expected after the Horiba instrument is calibrated.

After calibration is confirmed, the same calibration gas will be used to fill a 3 liter Tedlar® gas sampling bag. The customized sampling system (Figure 3-1) will be used to complete the calibration as follows:

Step 1. Disconnect all quick connect pneumatic fittings from sampling system with the exception of the hose. This ensures the system is sealed from all points except through the hose.

Step 2. Visually check that the red drain separator O-ring (approximately 2-inches in diameter) is visible at the opening of the sample inlet port, which is located on the front of the Horiba and to the right of the screen display. Insert the male pneumatic fitting on the end of the Horiba sampling tube to the female quick connect on the sampling system and ensure a secure fit.

Step 3. Ensure that the sampling system is purged according to the steps listed below under Section 3.2.1.2 - Cross Contamination Purging for Sampling System.

Step 4. Once within the given values, attach the Tedlar® bag to the male pneumatic fitting at the end of the braided stainless steel tubing.

Step 5. Open the Tedlar® bag valve to allow the calibration gas to be pulled through the sampling system.

Step 6. Record the instrument read-outs when the instrument has stabilized and compare the results to the calibration gas concentrations.

The mid-day calibration check will be performed using the same technique as the "flow though" portion of initial calibration described in steps one through six above.

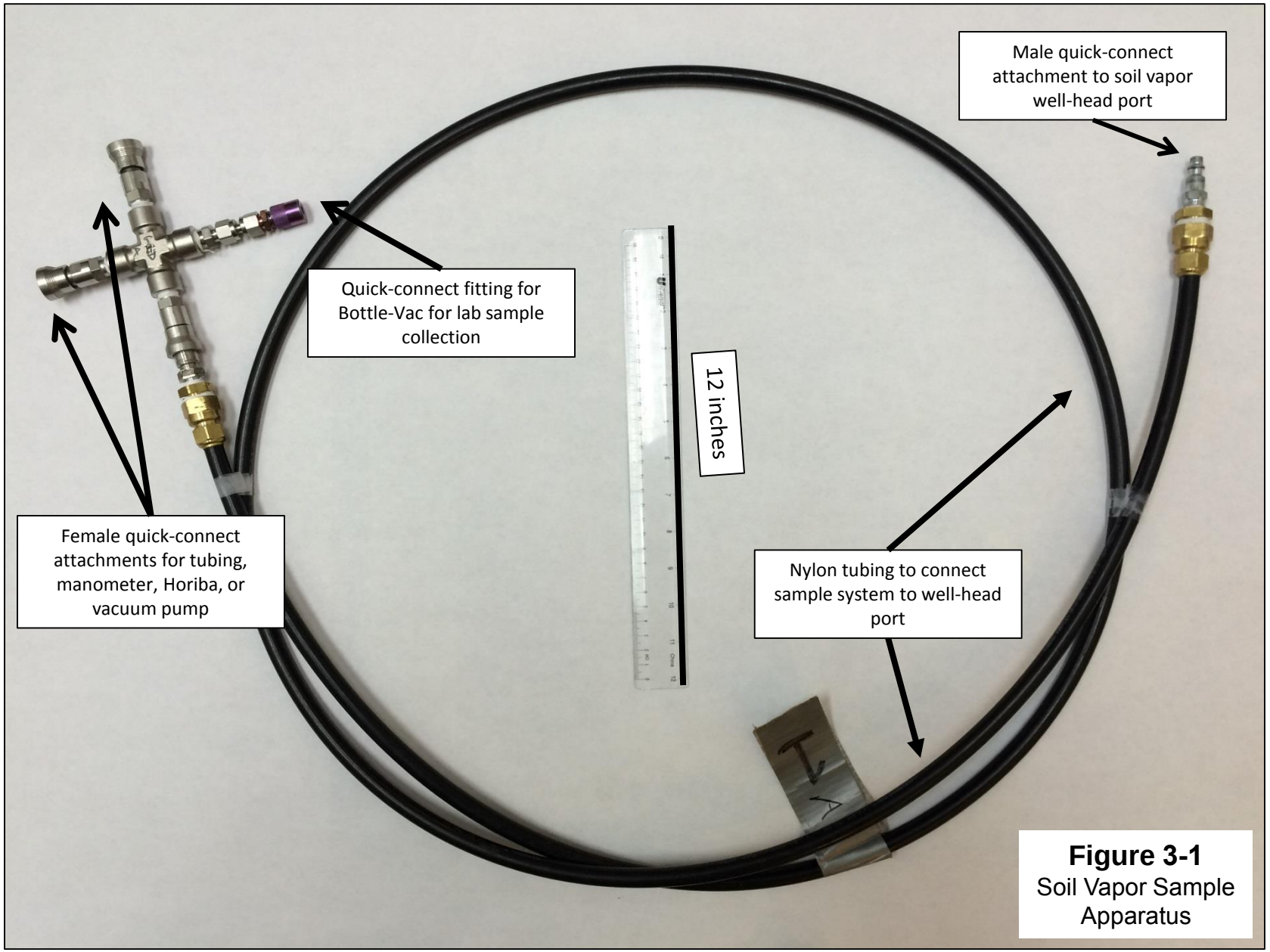


Figure 3-1
Soil Vapor Sample Apparatus

If the values for HC, O_2 and CO_2 are within 5% of the calibration values made using the calibration port, the calibration process is complete. As stated previously, the Horiba read-outs for HC are reported as hexane. A value of approximately 800 ppmv HC is expected. All other gas composition values should match the calibration gas values. If values are outside of this range, perform a leak check as described in Section 3.2.1.3 and follow the calibration process again.

If at any point during sampling, a reading for HC, O_2 or CO_2 reaches an unreasonable value (e.g., an O_2 concentration greater than 22%) or if a data value falls outside the trend indicated by previous readings at a given SVMP, a calibration check will be triggered. The expected range of values are: for HC - from 0 to 40,000 ppmv, for percent O_2 from 0 % to 22%, and for percent CO_2 from 0% to 15%. If any readings are outside of these ranges, a calibration check must be made and if necessary, the instrument will be recalibrated.

3.2.1.2 Cross Contamination Purging for Sampling System

The sampling system must be purged with ambient air before being attached to a SVMP sample port to minimize the potential for cross contamination between sample collections. To ensure the entire sample train is thoroughly purged, attach the pump to the setup and flush atmospheric air through the quick connect port and the nylon tubing. All quick connect pneumatic fittings are to be opened during this process by placing a male fitting into the female fitting to allow for flow. Monitor the purging effectiveness using the Horiba to ensure no contaminants are still present and only ambient air is being read. Correct values for ambient air must be less than 5 ppmv HC, between 20% to 22% for percent O_2, and 0% for percent CO_2. Complete instrument purging must be performed after sampling each SVMP.

3.2.1.3 Leak Check of Sample System

At the beginning of each day, the sampling system will be leak checked by using the pump to apply vacuum to the sampling system as follows:

Step 1. Cap the male pneumatic fitting on the end of the nylon tubing with a spare female quick disconnect fitted to a vacuum/pressure gauge.

Step 2. Connect the SVMP purging/sampling pump to one of the quick disconnect fittings on the sample system and evacuate the air from the sample system to establish a vacuum.

Step 3. Disconnect the pump and immediately record the vacuum reading from the pressure/vacuum gauge.

Step 4. After 10 minutes have elapsed, check and record the vacuum reading on the gauge.

Step 5. Verify that the starting and ending vacuum readings are within 10% to ensure that the sampling system is not leaking.

Step 6. If the two vacuum readings are not within 10% of each other, check the conditions of the seals and repeat the leak test until the sampling system is confirmed to be air tight.

3.2.2 Soil Vapor Sampling Procedures

3.2.2.1 Sample Train Setup

The Horiba analyzer must be turned on, warmed up, and calibrated according to the steps stated above and then attached to the sampling system. The Horiba analyzer is turned on for the first time at the beginning of the day and remains in the on position throughout the day. The Horiba analyzer is plugged

into the 12V DC outlet in the project vehicle using an AC inverter. All other equipment is gas powered or will be powered by generator, and can be powered off between sampling at each well. The pump is attached and sealed to the setup by a quick connect fitting. It is important that no pneumatic fittings besides the tubing to the soil vapor well port are attached prior to turning on the pump.

3.2.2.2 Static Pressure Measurement

Before taking the static pressure reading, the manometer instrument must be zeroed to atmospheric pressure. The screen should read 0.00 inches of water column (in WC). After confirming that the manometer is zeroed, the following procedure is used to connect the sampling system to the SVMPs and measure the static (also called baseline) pressure, to assure readiness for purging and sampling:

Step 1. Connect the manometer to the quick connect on the side of the sampling system opposite of the Bottle-Vac™ sample collection port (see Figure 3-1).

Step 2. Verify that the manometer reads 0.00 in WC.

Step 3. Insert the male quick connect fitting on the end of the nylon tubing to the female quick disconnect fitting on the top of the SVMP and ensure a secure connection.

Step 4. Monitor the change in manometer readings over time and record the pressure/vacuum reading when the meter stabilizes.

Note: Static pressure readings have typically ranged from +2.00 in (pressure) to -12.00 in WC (vacuum) at each soil vapor well port.

3.2.2.3 Well Purging

Stagnant soil vapor is purged from the SVMP as follows:

Step 1. Tum on the SVMP sampling pump, verify the operation of the flow rotameter, and check for potential leaks as necessary.

Step 2. Consult the Purge Table (Table 3-1) for the initial purge volume.

Step 3. Connect the female quick disconnect on the terminal end of the sampling system to the male quick connect on the vacuum side of the soil vapor monitoring sampling pump and start timing the purge cycle. (Note: Use the flow rate on the rotameter and the pre-calculated purge volume to quickly calculate the purge time. The purge time is determined by the well port diameter, well depth, and rate of the pump; all of which are known before sampling with the exception of the flowrate. The amount of vapor needed to be removed is based on one well casing volume.)

Step 4. After adequately purging for the appropriate time, quickly disconnect the sampling system from the vacuum pump. (Note: The sampling system is to remain connected to the SVMP for the duration of sampling.)

Step 5. Allow the manometer reading to return to within 0.10 in. WC of the static pressure reading before moving to the next step in the sampling procedure.

3.2.2.4 Horiba Readings

Once the SVMP has been purged, the following procedure is used to take and record HC, O_2 and CO_2 measurements using the calibrated Horiba:

Step l. Ensure that the Horiba is turned on and functioning properly.

Step 2. Record the manometer reading.

Step 3. Insert the male quick connect fitting into the female quick connect fitting on the terminal side of the sampling system and ensure a tight connection.

Step 4. Observe the Horiba O_2 reading for stability or for a maximum of one minute, whichever comes first.

Step 5. Record the O_2, CO_2, and HC readings and quickly disconnect the Horiba. Photograph the Horiba reading for quality control (QC) reference. Include the well number in the picture.

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
1	KAFB-106028	KAFB-106028-150	148.75	151.25	0.50	0.00136	0.409	0.615
		KAFB-106028-250	248.75	251.25	0.50	0.00136	0.409	0.752
		KAFB-106028-350	348.75	351.25	0.50	0.00136	0.409	0.888
		KAFB-106028-450	448.75	451.25	0.50	0.00136	0.409	1.024
2	KAFB-106108	KAFB-106108-025	15.30	25.30	0.75	0.00307	1.636	1.714
		KAFB-106108-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106108-150	140.20	150.20	0.75	0.00307	1.636	2.097
		KAFB-106108-250	240.30	250.30	0.75	0.00307	1.636	2.404
		KAFB-106108-350	340.30	350.30	0.75	0.00307	1.636	2.711
		KAFB-106108-450	440.00	450.00	3.00	0.04909	1.636	23.725
3	KAFB-106109	KAFB-106109-025	15.20	25.20	0.75	0.00307	1.636	1.713
		KAFB-106109-050	40.10	50.10	0.75	0.00307	1.636	1.790
		KAFB-106109-150	140.00	150.00	0.75	0.00307	1.636	2.096

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106109-250	240.20	250.20	0.75	0.00307	1.636	2.404
		KAFB-106109-350	340.60	350.60	0.75	0.00307	1.636	2.712
		KAFB-106109-450	440.00	450.00	3.00	0.04909	1.636	23.725
4	KAFB-106110	KAFB-106110-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106110-050	40.10	50.10	0.75	0.00307	1.636	1.790
		KAFB-106110-150	140.30	150.30	0.75	0.00307	1.636	2.097
		KAFB-106110-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106110-350	340.20	350.20	0.75	0.00307	1.636	2.710
		KAFB-106110-450	440.00	450.00	3.00	0.04909	1.636	23.725
5	KAFB-106111	KAFB-106111-025	15.20	25.20	0.75	0.00307	1.636	1.713
		KAFB-106111-050	40.10	50.10	0.75	0.00307	1.636	1.790
		KAFB-106111-150	140.30	150.30	0.75	0.00307	1.636	2.097
		KAFB-106111-250	240.30	250.30	0.75	0.00307	1.636	2.404
		KAFB-106111-350	340.40	350.40	0.75	0.00307	1.636	2.711
		KAFB-106111-450	440.30	450.30	3.00	0.04909	1.636	23.740
6	KAFB-106112	KAFB-106112-025	15.00	25.00	0.75	0.00307	1.636	1.713

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		4KAFB-1506112-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106112-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106112-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106112-350	339.00	349.00	0.75	0.00307	1.636	2.707
		KAFB-106112-450	439.00	449.00	3.00	0.04909	1.636	23.676
7	KAFB-106113	KAFB-106113-020	10.00	20.00	0.75	0.00307	1.636	1.697
		KAFB-106113-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106113-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106113-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106113-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106113-450	440.00	450.00	3.00	0.04909	1.636	23.725
8	KAFB-106114	KAFB-106114-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106114-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106114-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106114-250	235.00	245.00	0.75	0.00307	1.636	2.388
		KAFB-106114-350	340.00	350.00	0.75	0.00307	1.636	2.710

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106114-450	439.60	449.60	3.00	0.04909	1.636	23.706
9	KAFB-106115	KAFB-106115-025	14.60	24.60	0.75	0.00307	1.636	1.711
		KAFB-106115-050	39.60	49.60	0.75	0.00307	1.636	1.788
		KAFB-106115-150	144.60	154.60	0.75	0.00307	1.636	2.110
		KAFB-106115-250	239.60	249.60	0.75	0.00307	1.636	2.402
		KAFB-106115-350	339.60	349.60	0.75	0.00307	1.636	2.709
		KAFB-106115-450	439.60	449.60	3.00	0.04909	1.636	23.706
10	KAFB-106116	KAFB-106116-025	10.00	19.45	0.75	0.00307	1.546	1.606
		KAFB-106116-050	40.00	49.45	0.75	0.00307	1.546	1.698
		KAFB-106116-150	140.00	149.45	0.75	0.00307	1.546	2.005
		KAFB-106116-250	240.00	249.45	0.75	0.00307	1.546	2.311
		KAFB-106116-350	340.00	349.45	0.75	0.00307	1.546	2.618
		KAFB-106116-450	440.00	448.95	3.00	0.04909	1.464	23.502
11	KAFB-106117	KAFB-106117-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106117-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106117-150	140.00	150.00	0.75	0.00307	1.636	2.096

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106117-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106117-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106117-450	440.00	450.00	3.00	0.04909	1.636	23.725
12	KAFB-106118	KAFB-106118-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106118-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106118-160	150.00	160.00	0.75	0.00307	1.636	2.127
		KAFB-106118-265	255.00	265.00	0.75	0.00307	1.636	2.449
		KAFB-106118-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106118-450	440.00	450.00	3.00	0.04909	1.636	23.725
13	KAFB-106119	KAFB-106119-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106119-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106119-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106119-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106119-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106119-450	440.00	450.00	3.00	0.04909	1.636	23.725
14	KAFB-106120	KAFB-106120-025	15.00	25.00	0.75	0.00307	1.636	1.713

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106120-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106120-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106120-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106120-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106120-450	434.00	444.00	3.00	0.04909	1.636	23.431
15	KAFB-106121	KAFB-106121-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106121-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106121-145	135.00	145.00	0.75	0.00307	1.636	2.081
		KAFB-106121-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106121-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106121-440	434.00	444.00	3.00	0.04909	1.636	23.431
16	KAFB-106122	KAFB-106122-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106122-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106122-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106122-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106122-350	340.00	350.00	0.75	0.00307	1.636	2.710

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106122-450	434.00	444.00	3.00	0.04909	1.636	23.431
17	KAFB-106123	KAFB-106123-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106123-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106123-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106123-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106123-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106123-450	432.00	442.00	3.00	0.04909	1.636	23.333
18	KAFB-106124	KAFB-106124-025	15.10	25.00	0.75	0.00307	1.620	1.696
		KAFB-106124-050	40.10	50.00	0.75	0.00307	1.620	1.773
		KAFB-106124-150	140.10	150.00	0.75	0.00307	1.620	2.080
		KAFB-106124-250	240.10	250.00	0.75	0.00307	1.620	2.387
		KAFB-106124-350	340.10	350.00	0.75	0.00307	1.620	2.693
		KAFB-106124-450	440.10	450.00	3.00	0.04909	1.620	23.709
19	KAFB-106125	KAFB-106125-025	15.20	25.00	0.75	0.00307	1.603	1.680
		KAFB-106125-050	40.20	50.00	0.75	0.00307	1.603	1.757
		KAFB-106125-150	140.20	150.00	0.75	0.00307	1.603	2.063

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106125-250	240.20	250.00	0.75	0.00307	1.603	2.370
		KAFB-106125-350	340.20	350.00	0.75	0.00307	1.603	2.677
		KAFB-106125-450	440.20	450.00	3.00	0.04909	1.603	23.693
20	KAFB-106126	KAFB-106126-025	15.10	25.00	0.75	0.00307	1.620	1.696
		KAFB-106126-050	40.10	50.00	0.75	0.00307	1.620	1.773
		KAFB-106126-150	140.10	150.00	0.75	0.00307	1.620	2.080
		KAFB-106126-250	240.10	250.00	0.75	0.00307	1.620	2.387
		KAFB-106126-350	340.10	350.00	0.75	0.00307	1.620	2.693
		KAFB-106126-450	440.20	450.00	3.00	0.04909	1.603	23.693
21	KAFB-106127	KAFB-106127-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106127-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106127-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106127-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106127-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106127-450	440.00	450.00	3.00	0.04909	1.636	23.725
22	KAFB-106128	KAFB-106128-025	15.04	25.04	0.75	0.00307	1.636	1.713

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106128-050	40.07	50.07	0.75	0.00307	1.636	1.790
		KAFB-106128-150	140.19	150.19	0.75	0.00307	1.636	2.097
		KAFB-106128-250	240.29	250.29	0.75	0.00307	1.636	2.404
		KAFB-106128-350	340.39	350.39	0.75	0.00307	1.636	2.711
		KAFB-106128-450	440.06	450.06	3.00	0.04909	1.636	23.728
23	KAFB-106129	KAFB-106129-025	15.10	25.10	0.75	0.00307	1.636	1.713
		KAFB-106129-050	39.70	49.70	0.75	0.00307	1.636	1.788
		KAFB-106129-150	140.20	150.20	0.75	0.00307	1.636	2.097
		KAFB-106129-250	240.10	250.10	0.75	0.00307	1.636	2.403
		KAFB-106129-350	337.40	347.40	0.75	0.00307	1.636	2.702
		KAFB-106129-450	440.70	450.70	3.00	0.04909	1.636	23.760
24	KAFB-106130	KAFB-106130-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106130-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106130-150	150.00	160.00	0.75	0.00307	1.636	2.127
		KAFB-106130-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106130-350	340.00	350.00	0.75	0.00307	1.636	2.710

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106130-450	440.00	450.00	3.00	0.04909	1.636	23.725
25	KAFB-106131	KAFB-106131-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106131-055	45.00	55.00	0.75	0.00307	1.636	1.805
		KAFB-106131-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106131-245	235.00	245.00	0.75	0.00307	1.636	2.388
		KAFB-106131-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106131-450	430.00	440.00	3.00	0.04909	1.636	23.234
26	KAFB-106132	KAFB-106132-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106132-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106132-175	164.00	174.00	0.75	0.00307	1.636	2.170
		KAFB-106132-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106132-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106132-450	440.00	450.00	3.00	0.04909	1.636	23.725
27	KAFB-106133	KAFB-106133-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106133-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106133-170	160.00	170.00	0.75	0.00307	1.636	2.158

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106133-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106133-350	339.00	349.00	0.75	0.00307	1.636	2.707
		KAFB-106133-450	439.00	449.00	3.00	0.04909	1.636	23.676
28	KAFB-106134	KAFB-106134-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106134-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106134-170	160.00	170.00	0.75	0.00307	1.636	2.158
		KAFB-106134-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106134-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106134-450	440.00	450.00	3.00	0.04909	1.636	23.725
29	KAFB-106135	KAFB-106135-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106135-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106135-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106135-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106135-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106135-450	440.00	450.00	3.00	0.04909	1.636	23.725
30	KAFB-106136	KAFB-106136-025	15.00	25.00	0.75	0.00307	1.636	1.713

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106136-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106136-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106136-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106136-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106136-450	440.00	450.00	3.00	0.04909	1.636	23.725
31	KAFB-106137	KAFB-106137-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106137-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106137-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106137-250	240.10	250.10	0.75	0.00307	1.636	2.403
		KAFB-106137-350	340.50	350.50	0.75	0.00307	1.636	2.711
		KAFB-106137-450	440.00	450.00	3.00	0.04909	1.636	23.725
32	KAFB-106138	KAFB-106138-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106138-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106138-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106138-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106138-350	340.00	350.00	0.75	0.00307	1.636	2.710

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106138-450	440.00	450.00	3.00	0.04909	1.636	23.725
33	KAFB-106139	KAFB-106139-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106139-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106139-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106139-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106139-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106139-450	440.00	450.00	3.00	0.04909	1.636	23.725
34	KAFB-106140	KAFB-106140-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106140-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106140-150	141.80	151.80	0.75	0.00307	1.636	2.102
		KAFB-106140-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106140-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106140-450	440.00	450.00	3.00	0.04909	1.636	23.725
35	KAFB-106141	KAFB-106141-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106141-050	50.00	60.00	0.75	0.00307	1.636	1.820
		KAFB-106141-170	160.00	170.00	0.75	0.00307	1.636	2.158

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106141-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106141-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106141-450	440.00	450.00	3.00	0.04909	1.636	23.725
36	KAFB-106142	KAFB-106142-030	20.00	30.00	0.75	0.00307	1.636	1.728
		KAFB-106142-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106142-170	160.00	170.00	0.75	0.00307	1.636	2.158
		KAFB-106142-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106142-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106142-450	440.00	450.00	3.00	0.04909	1.636	23.725
37	SVEW-01	SVEW-01-260	245.00	260.00	2.00	0.02182	2.454	8.126
38	SVEW-02/03	SVEW-02-060	45.00	60.00	2.00	0.02182	2.454	3.763
		SVEW-03-160	145.00	160.00	2.00	0.02182	2.454	5.945
39	SVEW-04/05	SVEW-04-313	298.00	313.00	2.00	0.02182	2.454	9.283
		SVEW-05-460	445.00	460.00	2.00	0.02182	2.454	12.490
40	SVEW-06/07	SVEW-06-060	45.00	60.00	2.00	0.02182	2.454	3.763
		SVEW-07-160	145.00	160.00	2.00	0.02182	2.454	5.945
41	SVEW-08/09	SVEW-08-260	245.00	260.00	2.00	0.02182	2.454	8.126
		SVEW-09-460	443.00	457.00	2.00	0.02182	2.290	12.261
42	SVMW-01	SVMW-01-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-01-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-01-250	250.70	253.20	0.50	0.00136	0.409	0.754
		SVMW-01-300	308.50	310.00	0.50	0.00136	0.245	0.668
43	SVMW-02	SVMW-02-050	50.00	52.50	0.50	0.00136	0.409	0.481

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		SVMW-02-100	97.00	99.50	0.50	0.00136	0.409	0.545
		SVMW-02-150	150.00	152.50	0.50	0.00136	0.409	0.617
44	SVMW-03	SVMW-03-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-03-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-03-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-03-300	300.00	302.50	0.50	0.00136	0.409	0.821
45	SVMW-04	SVMW-04-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-04-100	98.00	100.50	0.50	0.00136	0.409	0.546
		SVMW-04-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-04-300	297.50	300.00	0.50	0.00136	0.409	0.818
46	SVMW-05	SVMW-05-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-05-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-05-230	229.50	231.00	0.50	0.00136	0.245	0.560
		SVMW-05-290	287.50	290.00	0.50	0.00136	0.409	0.804
47	SVMW-06	SVMW-06-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-06-100	99.50	102.00	0.50	0.00136	0.409	0.548
		SVMW-06-252	252.00	254.50	0.50	0.00136	0.409	0.756
		SVMW-06-302	302.50	305.00	0.50	0.00136	0.409	0.825
48	SVMW-07	SVMW-07-050	49.50	52.00	0.50	0.00136	0.409	0.480
		SVMW-07-100	95.50	98.00	0.50	0.00136	0.409	0.543
		SVMW-07-150	147.50	150.00	0.50	0.00136	0.409	0.614
49	SVMW-08[2]	SVMW-08-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-08-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-08-250	250.00	252.50	0.50	0.00136	0.409	0.753
50	SVMW-09	SVMW-09-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-09-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-09-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-09-266	266.00	268.50	0.50	0.00136	0.409	0.775
51	SVMW-10	SVMW-10-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-10-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-10-150	150.00	152.50	0.50	0.00136	0.409	0.617

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		SVMW-10-250	250.00	252.50	0.50	0.00136	0.409	0.753
52	SVMW-11	SVMW-11-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-11-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-11-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-11-260	260.00	262.50	0.50	0.00136	0.409	0.767
53	SVMW-12/ SVEW-10	SVMW-12-150	150.00	152.50	0.50	0.00136	0.409	0.617
		SVMW-12-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-12-350	350.00	352.50	0.50	0.00136	0.409	0.890
		SVMW-12-450	450.00	452.50	0.50	0.00136	0.409	1.026
		SVEW-10-410	400.00	410.00	2.00	0.02182	1.636	10.581
54	SVMW-13/ SVEW-11	SVMW-13-150	150.00	152.50	0.50	0.00136	0.409	0.617
		SVMW-13-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-13-350	350.00	352.50	0.50	0.00136	0.409	0.890
		SVMW-13-450	450.00	452.50	0.50	0.00136	0.409	1.026
		SVEW-11-410	400.00	410.00	2.00	0.02182	1.636	10.581
55	SVMW-14/ SVEW-12	SVMW-14-150	150.00	152.50	0.50	0.00136	0.409	0.617
		SVMW-14-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-14-350	350.00	352.50	0.50	0.00136	0.409	0.890
		SVMW-14-450	450.00	452.50	0.50	0.00136	0.409	1.026
		SVEW-12-410	400.00	410.00	2.00	0.02182	1.636	10.581
56	SVMW-15/ SVEW-13	SVMW-15-150	150.00	152.50	0.50	0.00136	0.409	0.617
		SVMW-15-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-15-350	350.00	352.50	0.50	0.00136	0.409	0.890
		SVMW-15-450	450.00	452.50	0.50	0.00136	0.409	1.026
		SVEW-13-410	400.00	410.00	2.00	0.02182	1.636	10.581

[1]borehole casing factor = (((10 inch diameter/12 inches)^2)(3.14159/4))(0.3 porosity)= 0.1636

[2]SVMW-08-266 is clogged and cannot be sampled

3.2.2.5 Bottle-VacTM Sampling

The following procedure will be used when collecting Bottle-Vac™ samples for laboratory analyses:

Step 1. Check the vacuum in a Bottle-Vac™ prior to sampling by taking a vacuum reading using a lab-supplied vacuum gauge. The gauge is connected to the bottle through a quick connect set in the same way that the bottle is connected to the sampling system. (Note: Bottles are required to be within 10% of -26 inches of mercury (in Hg) of vacuum. If a lower vacuum is measured, do not use the Bottle-Vac™ for sample collection.)

Step 2. With the nylon tubing still connected to the well port, record the static pressure indicated on the manometer read-out.

Step 3. Connect the Bottle-Vac™ to the specialized female pneumatic connection on the sample system (see Figure 3-1).

Step 4. Disconnect the Bottle-Vac™ after two minutes or once the manometer reading returns to static pressure, whichever comes first.

Step 5. Check the vacuum in the Bottle-Vac™ after removing from the sampling system. (Note: The vacuum should read no higher than 0.10 in Hg of vacuum. If there is still a positive vacuum reading, re-attach the Bottle- Vac™ to the sampling system for two additional minutes before checking the pressure once more; repeat as necessary.)

Bottle-Vacs will be shipped weekly to ALS Environmental Laboratories (ALS) in Simi Valley California, where they will be analyzed for the following analytical methods:

- Volatile organic compounds (VOC) in air by modified method TO-15
- Ethylene dibromide (EDB) by California Air Resources Board method 422
- Air-Phase Petroleum Hydrocarbons (APH) by MA APH 1.0
- Fixed Gases (H_2, Carbon Monoxide, CO_2, N_2, CH_4 and O_2/Ar) by modified U.S. Environmental Protection Agency (EPA) Method 3C

3.2.2.6 Humidity Measurements

Field measurements of subsurface humidity may be collected during future soil vapor sampling events to evaluate the water activity potential in the vadose zone for the assessment of natural biodegradation conditions.

A humidity probe will be used to collect soil-gas humidity and temperature measurements following SVMP purging and sample collection. The humidity probe will be calibrated each day it is used following the manufacturer instructions. The probe will be kept at a steady temperature while being calibrated.
Once calibrated, the humidity probe will be connected to the SVM point. The probe end of the instrument will be sealed so that only soil-gas humidity is read. Once connected to the SVM point, the vacuum pump will be connected to the top of the flow-through chamber so that soil-gas is pulled past the probe. The instrument will not read as accurately in non-moving air. Both the temperature and relative humidity of the soil-gas will be recorded.

3.2.3 Annual Monitoring Network Maintenance

Operations and maintenance for the soil vapor monitoring network will be performed during the sampling events. Wellheads will be inspected for integrity and necessary repairs will be performed as soon as possible. The findings of the inspections and the repairs will be documented by photographs and on the appropriate field forms (Appendix A).

3.3 Drinking Water Production Well Sampling

Sundance will perform monthly sampling of four drinking water production wells: KAFB-3, KAFB-15, KAFB-16 and VA-2 (Figure 2-2). These existing drinking water production wells at KAFB and the VA Hospital actively provide drinking water to the facilities' employees and inhabitants. Because the wells will be actively producing water during sampling, water levels at these wells will not be measured prior to sampling. In addition, one well volume will not be purged prior to sampling. Sampling at the drinking water production wells will be performed in accordance with the following steps:

Step 1. A Yellow Springs Instruments (YSI) 556 multi-probe system multi-parameter instrument will be used to collect field readings for dissolved oxygen (DO), pH, oxidation reduction potential (ORP), conductivity, and temperature during sampling. Calibrate the YSI according to the manufacturer's instructions for pH, ORP, conductivity and DO. Record the readings in a calibration log. Turbidity will be measured using a portable turbidimeter.

Step 2. Decontamination of the YSI will take place before use at each drinking water supply well location. Decontamination will entail rinsing the YSI to remove contaminants of concern that may impact study objectives. Specifications for decontamination materials are as follows:

- Use a standard brand of phosphate-free laboratory detergent, preferably either liquid Liquinox® or powder Alconox®.
- Use bottled water for the wash. Soap and water will remove the gross contamination from the sampling equipment.
- Use deionized water for the final rinse of YSI probes and containers that have direct contact to the sampling medium.

Step 3. Place a bucket underneath the sample port at the wellhead, and open the sample port. Purge any water in the sample port for thirty seconds to ensure that any accumulated sediment is removed.

Step 4. Fill the lower container of the YSI from the sample port and take a baseline reading of DO, pH, ORP, conductivity, and temperature. Fill the sample cell of the portable turbidimeter and collect the turbidity reading. Record these parameters on the sample collection log.

Step 5. Fill the water sample containers in accordance with requirements of the QAPjP (Appendix C). Samples for volatile organic analysis will be collected first. The sample bottles will be carefully filled to avoid overflow and potential loss of preservative, and tapped so entrapment of air is minimized and no head space exists. If bubbles appear, the vial will be refilled or a new vial will be used if a sample preservative (e.g., hydrochloric acid) is used.

Step 6. Place analytical samples in a cooler and chill to 4 degrees Celsius (°C). Samples must be shipped to the appropriate laboratory within 24 hours. The sample cooler must be shaded from direct sunlight immediately after collection.

Step 7. The field logbook, sample log sheet, labels, custody seals, and chain-of-custody forms will be filled out during sample collection.

Drinking water production well samples will be shipped to ALS laboratory in Kelso, WA where they will be analyzed for the following analytical parameters:

- EDB by EPA Method 504.1
- Benzene, toluene, ethylbenzene, and xylenes (BTEX) by EPA Method 524.2

3.4 Soil Vapor Monitoring Well Abandonment

Soil vapor monitoring location and well abandonment techniques and details will be provided in future revisions of this WP. Once the monitoring location(s) to be abandoned has been determined, the specifications will be submitted describing requirements for abandonment.

3.5 Soil Vapor Monitoring Well Installation

Drilling techniques and details on the design and installation of the soil vapor monitoring location(s) will be provided in future revisions of this WP. Once the design of the SVMPs has been completed, then specifications will be submitted describing the installation requirements for these locations.

3.6 Equipment Decontamination

The objective of field decontamination is to remove contaminants of concerns from sampling, and other field equipment to concentrations that will not impact study objectives.

Decontamination procedures specific to soil vapor sampling are outlined in Section 3.2.1.2. It is not anticipated that any additional decontamination procedures will be required for soil vapor sampling.

Decontamination procedures specific to drinking water supply well sampling are outlined in Step 2 of Section 3.3. It is not anticipated that any additional decontamination procedures will be required for drinking water supply well sampling.

3.7 Personal Protective Equipment

Modified Level D PPE will be worn during sampling as described in the project APP, which was submitted under a separate cover. Please reference Section 9.0 of the APP for more detailed information on PPE.

3.8 Photoionization Detector

A PID will be used for breathing zone monitoring during sampling activities. The PID will be calibrated and tested as required in the QAPjP (Appendix C).

3.9 Field Quality Control

Field QC samples will be collected throughout field investigation activities to ensure the integrity and reproducibility of data. Field QC samples include duplicates and trip blanks for VOC analysis.

Field QC samples are discussed in the QAPjP (Appendix C) and are listed below:

- Field duplicate samples (water/vapor) – 10% of total number of environmental samples per event
- Matrix spike/matrix spike duplicate samples (water) – 5% of total number of environmental samples per event
- Trip blank samples (water/vapor) – one per each shipment of groundwater and vapor samples per event for VOCs only
- Temperature blank (water) – 1 per each shipment of environmental samples

3.10 Sample Packaging and Shipping

The primary objective of sample packaging and shipping requirements is to maintain sample integrity from the time a sample is collected until it is received at the analytical laboratory. Chain-of-custody forms, sample labels, custody seals, and other sample documents will be completed as specified in the QAPjP, provided in Appendix C. Specific procedures for packaging and shipping of environmental samples are presented below:

Step 1. A sample label is attached to the sample bottle and completed with indelible ink.

Step 2. For water samples, a cooler (such as a Coleman or other sturdy cooler) will be used as a shipping container. In preparation for shipping samples, the drain plug will be taped shut so that no fluids, such as melted ice, will drain out of the cooler during shipment. A large plastic bag may be used as a liner for the cooler and packing material, such as bubble wrap, or Styrofoam beads, will be placed in the bottom of the liner. All water samples for chemical analysis must be shipped cooled to 4 °C with ice. All samples will require icing prior to shipping.

Step 3. Soil vapor samples will be returned to the lab in the sample container boxes in which they were sent. There are no temperature or preservative requirements for shipping of the soil vapor samples.

Step 4. The liner will be taped closed, if used, and sufficient packing material will be used to prevent sample containers from making contact or rolling around during shipment.

Step 5. A copy of the completed chain-of-custody record will be placed inside the cooler or box.

Step 6. The cooler or box will be closed and taped shut with packing tape.

Step 7. Custody seals will be placed on the cooler or box.

Step 8. The cooler or box will be shipped in accordance with the particular sample media and corresponding hold times.

3.11 Investigation-Derived Waste

It is not anticipated that any investigation-derived waste (IDW) will be generated during soil vapor sampling.

In addition, all groundwater generated during drinking water supply well sampling events will be 100% captured and contained. Fluids purged or generated at the wellheads will be placed, to the acceptable filling capacity, in 5-gallon buckets that are secured to a truck bed and upon conclusion of the work day, will be discharged to the GWTS through the sump in the building floor. Prior to discharge to the GWTS, sample documentation will be provided to the GWTS operator demonstrating that the water to be discharged has two consecutive preceding sampling events documenting no contaminants meet the definitions of characteristic hazardous waste (40 Code of Federal Regulations [CFR] Part 261). If any previous sampling data are reported above the concentrations stated in 40 CFR Part 261, the IDW will be stored in a 90-day accumulation area where the drums will be labeled and pending laboratory analytical results. All water IDW and decontamination water from equipment cleaning across all site activities will be considered non-hazardous water.

The storage containers will not be left unattended. The quantity of water purged from each well and the total quantity of water transferred to the GWTS will be recorded. A minimal amount of fines are anticipated to be present in this water and pre-filtering before batching into the GWTS is not anticipated. If for any reason the GWTS cannot accept the purge water as it is generated (e.g., shut down for maintenance), the water will be temporarily stored in the IDW area on pallets and properly labeled until it can be discharged to the GWTS sump.

Non-reusable PPE will be disposed of in accordance with the project APP (Section 9.0). Any additional waste associated with sampling (plastic bags, paper waste, etc.) will be collected and disposed of via the COA waste management system.

IDW management details pertaining to any well installation or abandonment that may be performed will be provided in Section 7 of future revisions of this WP. Once the drilling techniques and waste streams are determined, specifications will be submitted describing the management of that waste.

3.12 Reporting

Analytical data collected during soil vapor and drinking water monitoring activities described in this WP will be included in the Periodic Monitoring Reports delivered to NMED (quarterly, or the most current NMED-approved reporting requirement) as part of the investigation at SWMU ST-106/SS-111.

First, second and third quarter CY reports will document the monitoring activities performed during each quarter and will provide the detailed information listed below. The fourth quarter CY report may have additional requirements or information, and include cumulative information from the entire year.

- Descriptions of field activities performed during the quarter
- Tables of analytical concentrations
- Maps illustrating contaminant concentrations at specific well locations

- Analytical laboratory data
- Data validation summary of laboratory data and discussion of data quality

4 PROJECT SCHEDULE

The project schedule is provided in Appendix B of this WP.

5 ORGANIZATIONAL PLAN

The organizational structure of the Sundance Team is shown on Figure 5-1. Table 5-1 summarizes the responsibilities, qualifications, and authorities of project team members.

Figure 5.1. Project Team Organization
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

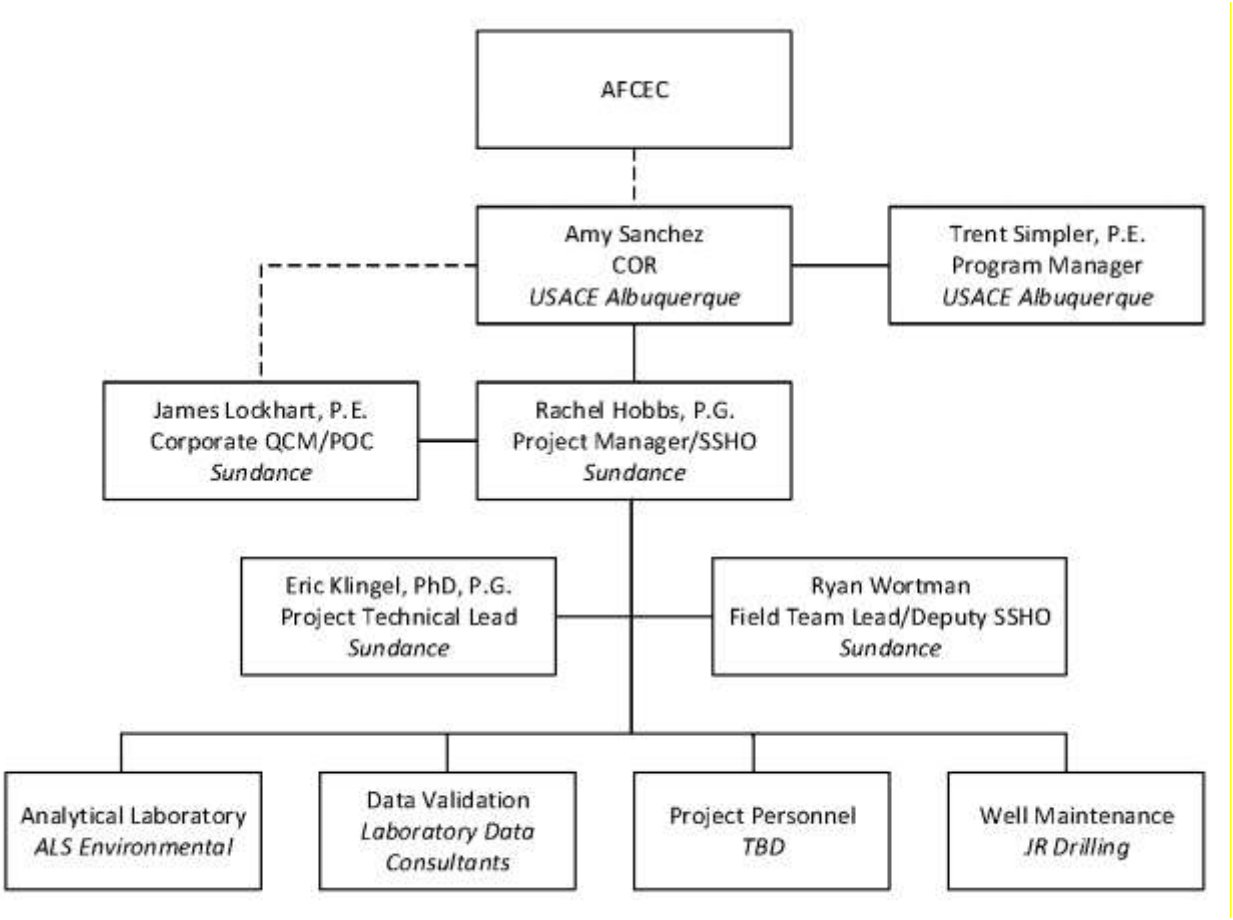


Table 5-1. Staff Roles, Responsibilities, and Authorities
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Position/Staff	Qualifications	Responsibilities	Authority Level
Vice President of Operations Jim Lockhart, P.E.	• BSME, MBA; • 33 years' experience in environmental remediation and engineering; • 25 years in management of environmental and engineering projects.	• As a Sundance Officer, authorized to negotiate and commit resources; • Primary point-of-contact for USACE on contractual and programmatic items; • Ensures consistency in deliverables and cost/performance reporting and progress reporting/invoicing; • Coordinates issue resolution as needed with Contracting Officer's Representative and/or Contracting Officer.	• Coordinates corrective action at programmatic level.
Project Manager/Site Safety and Health Officer Rachel Hobbs, P.G.	• M.S. in Geology; • Registered Professional Geologist in the state of Tennessee; • 5 years' experience in environmental remediation; • Past experience coordinating Kirtland BFF project tasks.	• Ensures that all work is accomplished with adequate internal controls; • Main point of contact for USACE on project-specific matters; • Reviews/confirms technical approach from kickoff meeting and throughout project execution to ensure project objectives are met; • Assembles and schedules resources; • Ensures on-schedule and high-quality services are delivered within budget; • Manages subcontractors;	• Full responsibility and authority to execute Task Orders; • Approves subcontractors' invoices, project charges, and deliverables; • Implements corrective action; • Stops work for any reason related to the project. • Approves APPs/SSHPs and all modifications before issuance to USACE; • Manages Health and Safety Program and directs training and required attendance;

Table 5-1. Staff Roles, Responsibilities, and Authorities
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Position/Staff	Qualifications	Responsibilities	Authority Level
		• Coordinates Sundance's participation in the Identifies and mitigates risks related to execution of the technical aspects of the work and ensures site safety; • Ensures work is performed in accordance with USACE/U.S. Air Force Guidelines, state/federal regulations; • Applies lessons learned from current and past projects; • Responsible for front and back end transition activities to ensure continuity on the project; • Ensures public relations sensitivities are met.	• Investigates safety concerns raised by staff; • Investigates any accidents;
Project Technical Lead Eric Klingel, PhD, P.G.	• PhD. in Geology; • Registered Professional Geologist in the state of North Carolina and South Carolina; • 30 years' experience in environmental site characterization, remediation and project management.	• Reports to the Project Manager and serves as the Alternative Project Manager; • Overall responsibility for design, implementation, and management of sampling activities; • Reviews all work plans, reporting, and data deliverables; • Coordinates with Field Personnel for oversight and QC; • Responsible for providing input for the design of the corrective actions	• Stops work for any reason including noncompliance /safety violation, or quality violations.

Table 5-1. Staff Roles, Responsibilities, and Authorities
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded)

Position/Staff	Qualifications	Responsibilities	Authority Level
		and reviews corrective elements specific to sampling; • Oversees development of APP in accordance with Engineer Manual 385-1-1 and Occupational Safety and Health Administration regulations; • Assists Project Manager and procurement staff in verification of safety performance of subcontractors Investigates any incidents, accidents, or safety violations Performs safety audits; • Manages monitoring reports.	
Field Team Lead/Deputy Site Safety and Health Officer Ryan Wortman	• B.S. in Geology; • Past experience coordinating Kirtland BFF project tasks.	• Reports to Technical Lead and/or Project Manager; • Oversees sampling team and sampling activities; • Coordinates with the Project Manager and Project Technical Lead on any deviations from the QAPjP due to changed field conditions such that data quality objectives are met; • Coordinates with SSHO to ensure that project activities are being performed in accordance with the APP. • Performs Health and Safety oversite in SSHO's absence	• Investigates safety concerns raised by staff; • Stop sampling work at any time due to safety or quality violations.

BFF – Bulk Fuels Facility
B.S. – Bachelor of Science Degree
M.S. – Master of Science Degree
P.E. – Professional Engineer
P.G. – Professional Geologist
QAPjP – Quality Assurance Project Plan
SSHO – Site Safety and Health Officer
SSHP – Site Safety and Health Plan
USACE – US Army Corps of Engineers

6 DATA MANAGEMENT PLAN

Environmental laboratory services will be provided only by laboratories compliant with the *DoD Quality Systems Manual for Environmental Laboratories, Version 5.0* (DoD, 2013) or a most recent version and that hold a current DoD Environmental Laboratory Accreditation Program accreditation for all appropriate analytical methods (DoD, 2013). ALS will provide analytical results in support of this project. ALS will provide electronic data in the Environmental Resource Program Information Management System (ERPIMS) format. The ERPIMS deliverable will be validated for upload to the U.S. Air Force (Air Force) data repository. All analytical data generated in support of this project will be uploaded to the Air Force Data Repository.

Analytical data generated in support of this project will undergo an EPA level III data review by Laboratory Data Consultants, Inc. (LDC). Automated data review software, developed by LDC, will be used to perform 100% EPA Level III data review. The data review will be performed for the monthly drinking water supply well data, as well as soil vapor analytical data obtained from each of the quarterly monitoring events. The data review will be performed using the QC criteria specified in Section 4.0 of the project QAPjP (Appendix C).

ERPIMS Version 5.0 submittals will be reviewed for accuracy and completeness before submittal. ERPIMS submittals will be provided to the Air Force, at a minimum, every six months or as appropriate for data generation for uploading to the Air Force data repository. Submittals will be deemed complete upon receipt of the insertion letter from the Air Force.

All project-related data will be maintained and archived in the electronic project files on the corporate server and will be made available to the government as necessary. All data generated in support of this contract will be maintained in accordance with the contract requirements.

7 INVESTIGATION-DERIVED WASTE MANAGEMENT PLAN

Additional project activities that require an IDW management plan may be required as part of this project. If any such project activities are preformed, an IDW management plan will be included in this section as part of a revision to this WP.

8 REFERENCES

DoD. 2013. *DoD Quality Systems Manual for Environmental Laboratories, Version 5.0.* July.

New Mexico Environment Department (NMED). 2010. Hazardous Waste Treatment Facility Operating Permit, EPA ID No. NM9570024423, issued to U.S. Air Force for the Open Detonation Unit Located at Kirtland Air Force Base, Bernalillo County, New Mexico, by the NMED Hazardous Waste Bureau. July.

Sundance Consulting, 2016. *Quarterly Soil Vapor Sampling and Monthly Drinking Water Sampling Accident Prevention Plan*. Sundance Consulting, Inc., February 2016.

USACE, 2011. *Vadose Zone Investigation Work Plan Bulk Fuels Facility Spill Solid Waste Management Units ST-106 and SS-111.* Prepared by Shaw Environmental and Infrastructure, Kirtland Air Force Base, New Mexico. March.

Appendix A
Field Forms

Field Activity Log

Job Number:	Task Description:	Date:

Weather:	Important Notes:	Sundance Employes Onsite:	Visitors:

Name:	Signature:	Date:

Field Activity Log (Continuation)

Page ____ of _____

Job Number:	Task Description:	Date:

Sundance Consulting Inc.

Well Integrity Checklist

Well ID: _____ **Inspector's Name:** _____
Date: _____
Time: _____ **Inspector's Signature:**_____

Before Opening Well

1. Is well cement pad in good condition? _____
2. Is lid securely tightened to vault?_____
3. Is well clearly labeled? _____
4. Do wells outside of BFF have security bolts? _____
5. Photograph well.

After Removing Lid Before Sampling Well

1. Is gasket worn or damaged? _____
2. Is vault flooded? _____
3. Are ports capped/labeled? _____
4. Are ports angled correctly? _____
5. Are all fittings and quick connects intact and operational?

   _____
6. Can you hear well breathing? _____
7. Photograph well with lid off.

During Sampling

1. Do all quick connects fit securely to sample system? _____
2. Does static pressure after purging return to initial static pressure within one minute?

   _____
3. Is well clogged? _____

Comments:_____

_____

Horiba Calibration Sheet

Horiba ID #: _____________

	Know Gas Mixture			Initial Horiba Reading			Post Cal. Horiba Readings			Flow Through Readings			
Date/Time	C3H8(ppm)	% CO2	% O2*	HC(ppm)	% CO2	% O2*	HC(ppm)	% CO2	% O2*	HC(ppm)	% CO2	% O2*	Initals
	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										
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	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										

* = Atmospheric %O2 Concentrations

Leak Test Log

Date	Intital Vacuum Reading	Time	Vacuum Reading After 10 Min.*	Time	Initials

*Min Minutes

Sundance Consulting Inc.

Purge Log

Project Name: Bulk Fuels Facility

Project Location: Kirtland Air Force Base

Well ID/Port Depth: _____

Samplers: _____

Sampler Signature/Date :_____

Screened Interval: Top:_____(ft. b.g.s)
Bottom:_____(ft. b.g.s)

Is Well damaged/Flooded: Yes - No

If yes, describe: _____

Weather Observations _____

Pump ID:_____ Horiba ID: _____ Sample System ID: _____ Manometer ID: _____

Was the sample system purged of hydrocarbons before connection with well port: Yes - No

Initial Static Pressure (inWC) _____

A. Pre- Calculated purge volume (cu. ft) Located on well data sheet	(Ft^3)
B. Flow Rate (SCFM) On flowmeter for air pump	(SCFM)
C. Purge time (Min. and Sec.) 1. Equals A/B 2. Write down whole number as minute 3. Multiple decimal by 60 for seconds	

Post Purge Static Pressure (inWC) _____

Sample Date/Time	CO2 %	O2%	Actual Purge Time	Hydrocarbons (ppmv)

Comments: _____

Bottle Vac. Number:_____

Initial B.V. Pressure:_____

Sample ID: _____

Final B.V. Pressure: _____

Reviewed by (Name): _____

Reviewer (Signature/Date): _____

Sundance Consulting Inc.

Date: _____

Time: _____

Project No.: __US01-023_____

C0C #: _____

Sample Collection Log

Project Name: ___KAFB_Bulk Fuels Facility_____

Sample No.: _____

Sample Location: ___Kirtland AFB ____

Sample Type: ____GRAB_____

Composite: (Y/N) _____No_____

Sample Team: _____

Trip Blank: _____

Sample:

Analytical Suite	Preservative	Container	TAT	Initials
VOCs by TO-15, EDB by CARB 422, Air-Phase Petroleum Hydrocarbons by MA APH 1.0, and Fixed Gases (H2, CO, CO2, N2, CH4 and O2/Ar) by E3C.	None	One Liter Amber Glass	15 Days	

Comments:

_____

_____

_____

_____

Logged By: _____ **Reviewed By**: _____

Air - Chain of Custody Record & Analytical Service Request

Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

COC #:

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

Company Name & Address (Reporting Information)

Sundance Consulting, Inc.
6700 Jefferson St NE, Suite C-3,
Albuquerque, NM 87109

Project Name/ Number: KAFB Bulk Fuels Facility/US01-023

Waynbill Number:

Project Manager: Rachel Hobbs

P.O. # / Billing Information:

Phone: 505-835-7660

Fax: 505-345-0742

Email Address for Result Reporting: rhobbs@sundance@sundance-inc.net

Sampler (Print & Sign):

Analytical Methods:

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Bottle Vac ID (Bar code # - AC, SC, etc.)	Flow Controller ID (Bar code # - FC #)	Bottle Vac Start Pressure "Hg	Bottle Vac End Pressure "Hg/psig	Sample Volume	TO-15 Project Specific List	MA APH HC Ranges	CARB 422 1,2-Dibromoethane	EPA 3C (O2, N2, CO2, CH4)	Comments e.g. Actual Preservative or specific instructions

Report Tier Levels - please select

Tier I - Results (Default if not specified) _____
Tier II (Results + QC Summaries) _____
Tier III (Results + QC & Calibration Summaries) _____
Tier IV (Data Validation Package) 10% Surcharge _____

EDD required Yes / No
Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:

Cooler / Blank Temperature ____°C

Sundance Consulting Inc.

Sample Collection Log

Date: _____

Time: _____

Project No: __US01-023_____

COC #:_____

Task:_____

Project Name ___KAFB Bulk Fuels Facility_____

Sample No. _____

Sample Location ___Kirtland AFB _____

Sample Type ____GRAB_____

Composite: Y/N ____No_____

Sample Team _____

Trip Blank _____

Sample:

Analytical Suite	Preservative	Quantity	Container	Temp.	FLT.	TAT	Initials
EDB by EPA 504.1	None	3	3x40 ml VOA	4°C	N	10 Days	
BTEX by EPA 524.2	HCL	3	3x40 ml VOA	4°C	N	10 Days	

Comments:

_____

_____

_____

_____

Logged by: _____ Reviewed by: _____

Groundwater- Chain of Custody Record & Analytical Service Request

Page _____ of _____

1317 S. 13th Ave
Kelso, WA 98626
Phone (360) 577 7222
Fax (360) 636 1068

Waybill #	COC #:

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

Company Name & Address (Reporting Information)	PO/Billing Information	Project Name / Project Number
Sundance Consulting, Inc. 6700 Jefferson St NE, Suite C-3, Albuquerque, NM 87109		KAFB Bulk Fuel Facilities US01-23
Project Manager: Rachel Hobbs	Sampler Name/Signature	Analytical Methods:
Phone: 505-835-7660 / Fax: 505-345-0742		
Email Address for Result Reporting: rhobbs@sundance@sundance-inc.net		

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Sample Matrix	No. Of Containers	Preservative					Sample Volume	Comments (e.g. specific instructions)

Report Tier Levels - please select

Tier I - Results (Default if not specified) _____
Tier II (Results + QC Summaries) _____
Tier III (Results + QC & Calibration Summaries) _____
Tier IV (Data Validation Package) 10% Surcharge _____

EDD required Yes / No
Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

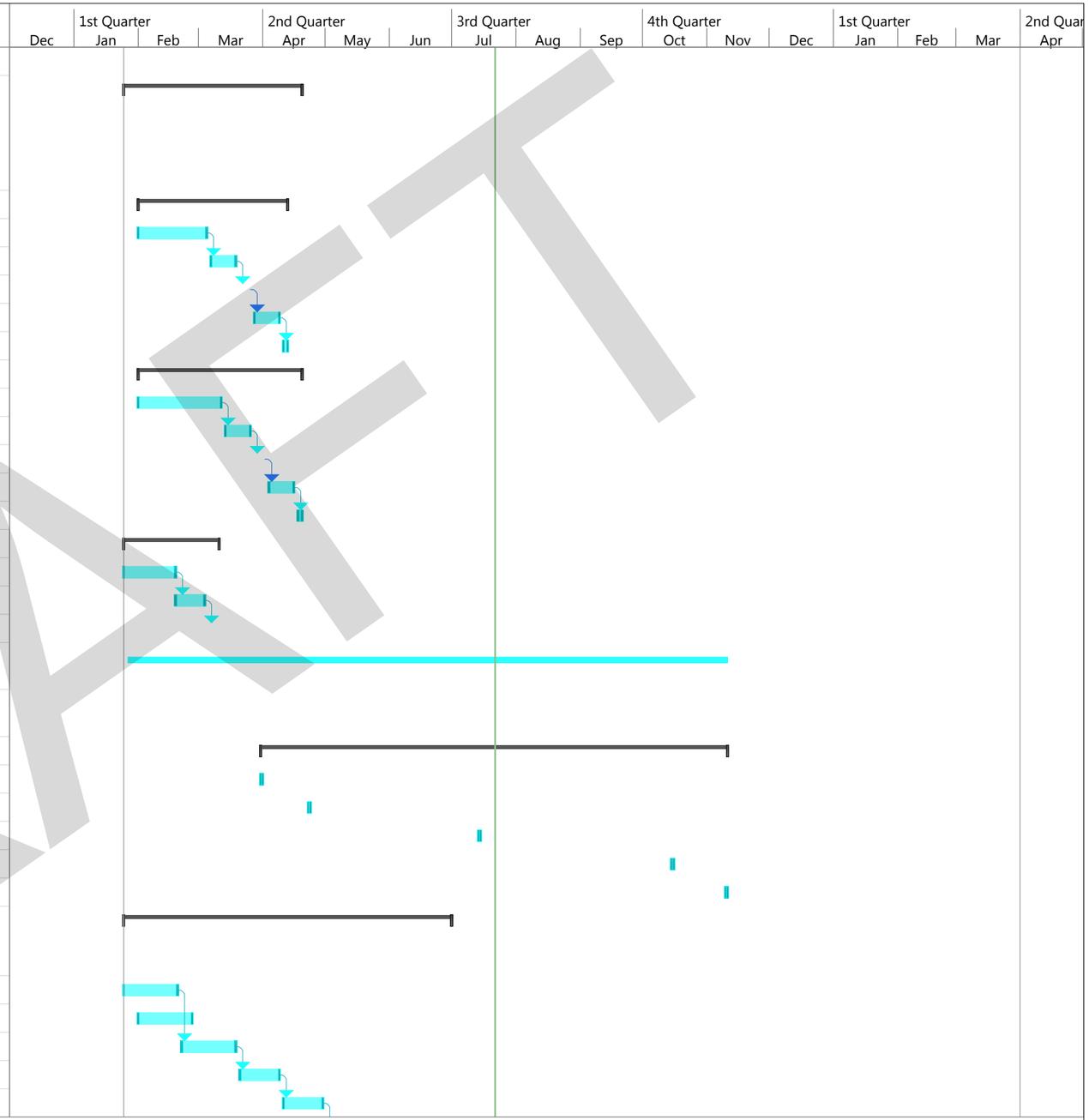
Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:		Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Date:	Time:		Received by: (Signature)	Date:	Time:

Cooler / Blank Temperature ____°C

Appendix B
Project Schedule

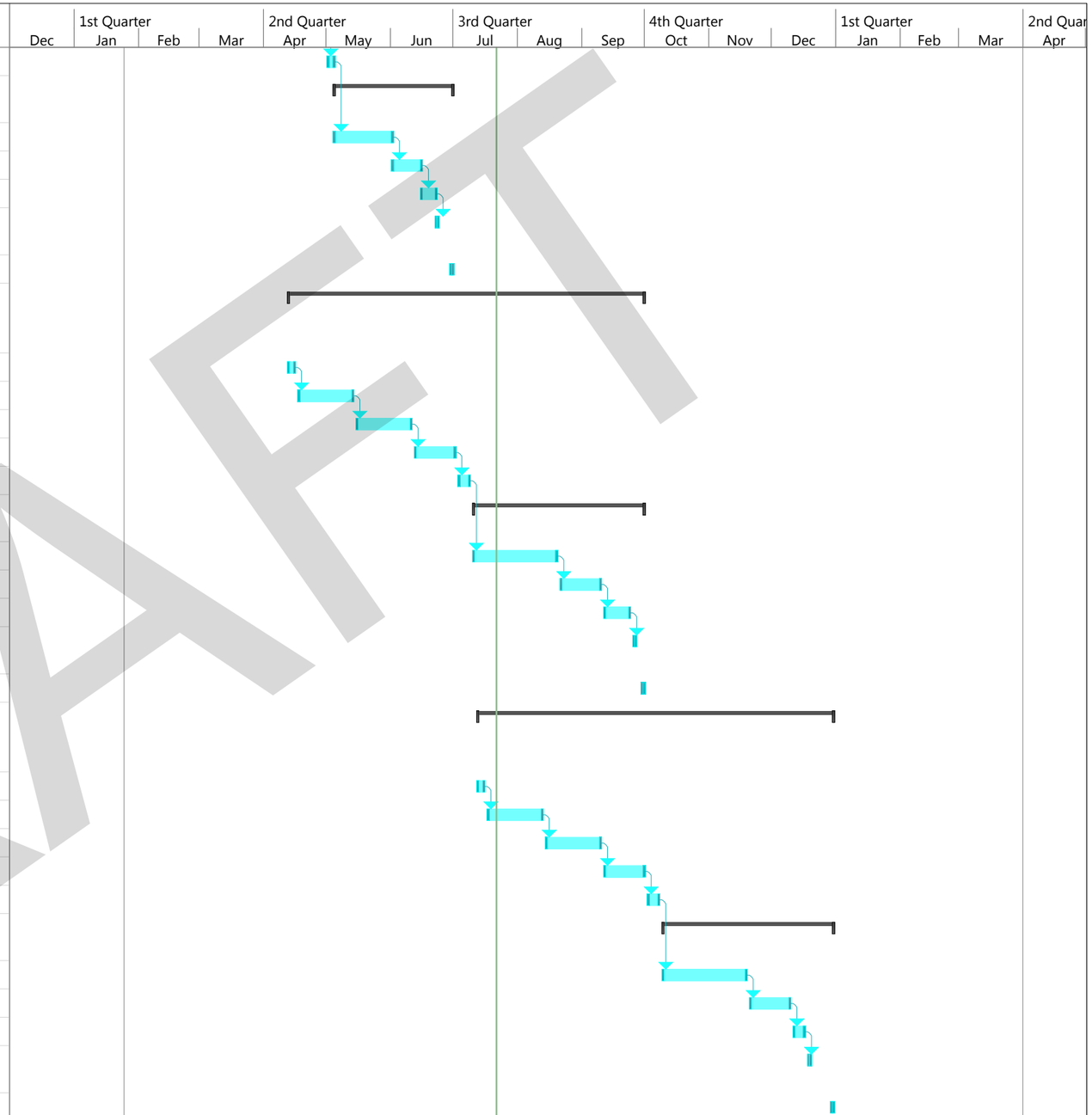
ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
1		Kick Off Meeting	1 day	Mon 2/1/16	Mon 2/1/16	
2		Update Quality Assurance Project Plan (Accident Prevention Plan (APP), Sampling and Analysis Plan (SAP), Field Sampling Plan (FSP)) CY 2016	62 days	Mon 1/25/16	Tue 4/19/16	
3		Work Plan	52 days	Mon 2/1/16	Tue 4/12/16	
4		Draft Work Plan	25 days	Mon 2/1/16	Fri 3/4/16	
5		USACE/AFCEC/KAFB Review	10 days	Mon 3/7/16	Fri 3/18/16	4
6		Incoporate Comments	5 days	Mon 3/21/16	Fri 3/25/16	5
7		Commander Signature	10 days	Mon 3/28/16	Fri 4/8/16	6
8		Delivery to NMED	2 days	Mon 4/11/16	Tue 4/12/16	7
9		QAPjP (Attached to Work Plan)	57 days	Mon 2/1/16	Tue 4/19/16	
10		Draft QAPjP	30 days	Mon 2/1/16	Fri 3/11/16	
11		USACE/AFCEC/KAFB Review	10 days	Mon 3/14/16	Fri 3/25/16	10
12		Incoporate Comments	5 days	Mon 3/28/16	Fri 4/1/16	11
13		Commander Signature	10 days	Mon 4/4/16	Fri 4/15/16	12
14		Delivery to NMED	2 days	Mon 4/18/16	Tue 4/19/16	13
15		APP	34 days	Mon 1/25/16	Thu 3/10/16	
16		Draft APP	19 days	Mon 1/25/16	Thu 2/18/16	
17		USACE/AFCEC/KAFB Review	10 days	Fri 2/19/16	Thu 3/3/16	16
18		Incoporate Comments	5 days	Fri 3/4/16	Thu 3/10/16	17
19		New Recurring Project Meetings CY 2016				
20		Status calls/CSI/other meetings to be held as needed				
21		Public Meetings CY 2016	161 days	Thu 3/31/16	Thu 11/10/16	
22		1st Public Meeting	1 day	Thu 3/31/16	Thu 3/31/16	
23		1st Field Trip	1 day	Sat 4/23/16	Sat 4/23/16	
24		2nd Public Meeting	1 day	Thu 7/14/16	Thu 7/14/16	
25		2nd Field Trip	1 day	Sat 10/15/16	Sat 10/15/16	
26		3rd Public Meeting	1 day	Thu 11/10/16	Thu 11/10/16	
27		Vadose Zone Sampling, Analysis and Reporting First Quarter CY 2016	114 days	Mon 1/25/16	Thu 6/30/16	
28		Project Mobilization	20 days	Mon 1/25/16	Fri 2/19/16	
29		Sample Planning	20 days	Mon 2/1/16	Fri 2/26/16	
30		Soil Vapor Sampling Field Work	20 days	Mon 2/22/16	Fri 3/18/16	28
31		Laboratory Analysis	15 days	Mon 3/21/16	Fri 4/8/16	30
32		Data Validation	15 days	Mon 4/11/16	Fri 4/29/16	31



Project: KAFB_Schedule_072216
Date: Fri 7/22/16

Task	Project Summary	Manual Task	Start-only	Deadline	
Split	Inactive Task	Duration-only	Finish-only	Progress	
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
33		Database Management	3 days	Mon 5/2/16	Wed 5/4/16	32
34		Data Dump Report First Quarter CY 2016	41 days	Thu 5/5/16	Thu 6/30/16	
35		Draft Data Dump Report	20 days	Thu 5/5/16	Wed 6/1/16	33
36		USACE/AFCEC/KAFB Review	10 days	Thu 6/2/16	Wed 6/15/16	35
37		Incoporporate Comments	5 days	Thu 6/16/16	Wed 6/22/16	36
38		Incorporation into Quarterly Report	1 day	Thu 6/23/16	Thu 6/23/16	37
39		Delivery to NMED	1 day	Thu 6/30/16	Thu 6/30/16	
40		Vadose Zone Sampling, Analysis and Reporting Second Quarter CY 2016	123 days	Wed 4/13/16	Fri 9/30/16	
41		Sample Planning	3 days	Wed 4/13/16	Fri 4/15/16	
42		Soil Vapor Sampling Field Work	20 days	Mon 4/18/16	Fri 5/13/16	41
43		Laboratory Analysis	20 days	Mon 5/16/16	Fri 6/10/16	42
44		Data Validation	15 days	Mon 6/13/16	Fri 7/1/16	43
45		Database Management	5 days	Mon 7/4/16	Fri 7/8/16	44
46		Data Dump Report Second Quarter CY 2016	60 days	Mon 7/11/16	Fri 9/30/16	
47		Draft Data Dump Report	30 days	Mon 7/11/16	Fri 8/19/16	45
48		USACE/AFCEC/KAFB Review	15 days	Mon 8/22/16	Fri 9/9/16	47
49		Incoporporate Comments	10 days	Mon 9/12/16	Fri 9/23/16	48
50		Incorporation into Quarterly Report	1 day	Mon 9/26/16	Mon 9/26/16	49
51		Delivery to NMED	1 day	Fri 9/30/16	Fri 9/30/16	
52		Vadose Zone Sampling, Analysis and Reporting Third Quarter CY 2016	123 days	Wed 7/13/16	Fri 12/30/16	
53		Sample Planning	3 days	Wed 7/13/16	Fri 7/15/16	
54		Soil Vapor Sampling Field Work	20 days	Mon 7/18/16	Fri 8/12/16	53
55		Laboratory Analysis	20 days	Mon 8/15/16	Fri 9/9/16	54
56		Data Validation	15 days	Mon 9/12/16	Fri 9/30/16	55
57		Database Management	5 days	Mon 10/3/16	Fri 10/7/16	56
58		Data Dump Report Third Quarter CY 2016	60 days	Mon 10/10/16	Fri 12/30/16	
59		Draft Data Dump Report	30 days	Mon 10/10/16	Fri 11/18/16	57
60		USACE/AFCEC/KAFB Review	15 days	Mon 11/21/16	Fri 12/9/16	59
61		Incoporporate Comments	5 days	Mon 12/12/16	Fri 12/16/16	60
62		Incorporation into Quarterly Report	1 day	Mon 12/19/16	Mon 12/19/16	61
63		Delivery to NMED	1 day	Fri 12/30/16	Fri 12/30/16	



Project: KAFB_Schedule_072216
Date: Fri 7/22/16

Task	Project Summary	Manual Task	Start-only	Deadline	
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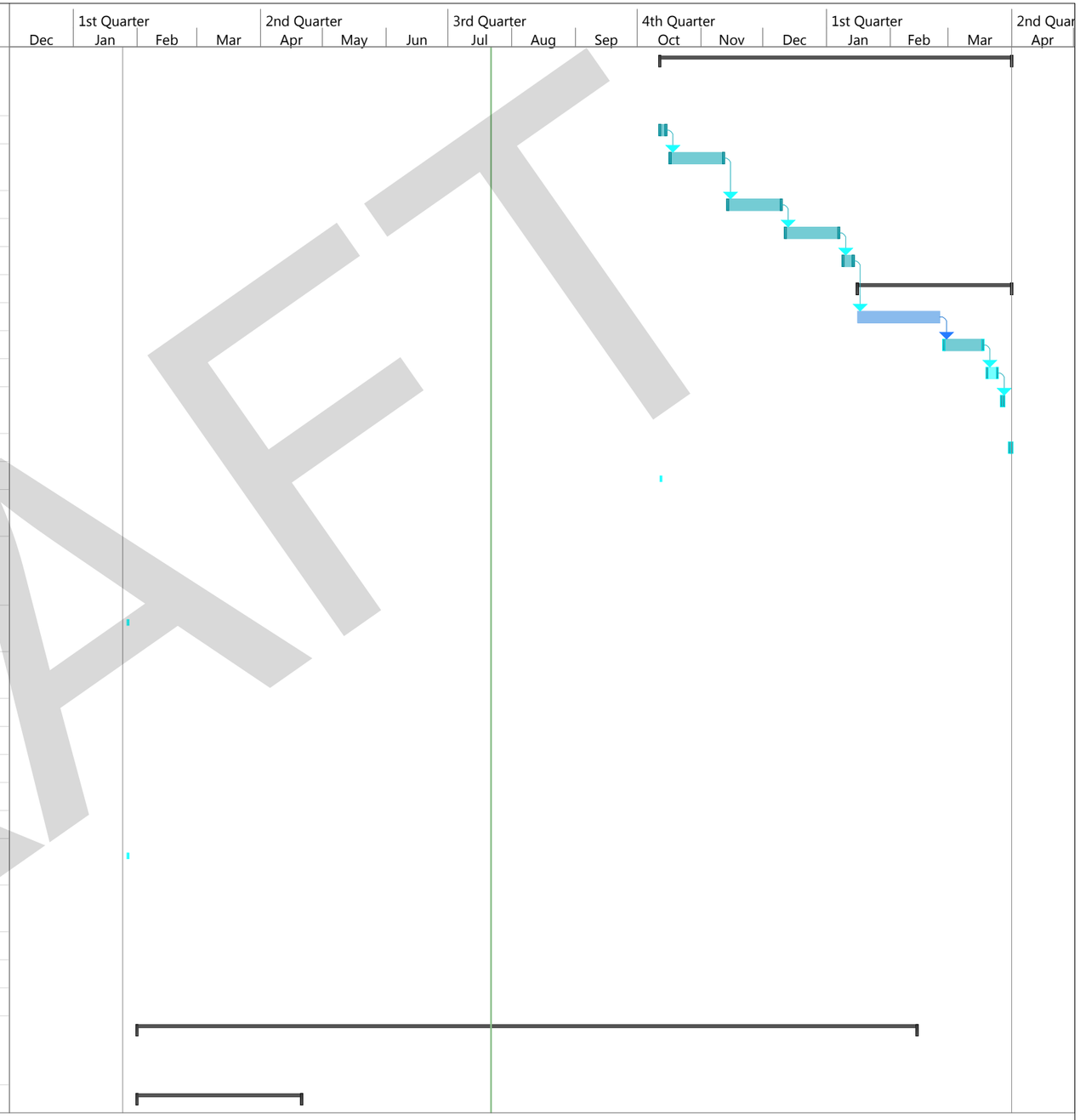
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
64		Vadose Zone Sampling, Analysis and Reporting Fourth Quarter CY 2016	123 days	Wed 10/12/16	Fri 3/31/17	
65		Sample Planning	3 days	Wed 10/12/16	Fri 10/14/16	
66		Soil Vapor Sampling Field Work	20 days	Mon 10/17/16	Fri 11/11/16	65
67		Laboratory Analysis	20 days	Mon 11/14/16	Fri 12/9/16	66
68		Data Validation	20 days	Mon 12/12/16	Fri 1/6/17	67
69		Database Management	5 days	Mon 1/9/17	Fri 1/13/17	68
70		Annual Monitoring Report	55 days	Mon 1/16/17	Fri 3/31/17	
71		Draft Annual Monitoring Report	30 days	Mon 1/16/17	Fri 2/24/17	69
72		USACE/AFCEC/KAFB Review	15 days	Mon 2/27/17	Fri 3/17/17	71
73		Incoporporate Comments	5 days	Mon 3/20/17	Fri 3/24/17	72
74		Incorporation into Quarterly Report	1 day	Mon 3/27/17	Mon 3/27/17	73
75		Delivery to NMED	1 day	Fri 3/31/17	Fri 3/31/17	
76		Maintenance Activities				
77		To be scheduled as determined necessary				
78		Well Installation and Abandonment - To be scheduled as determined necessary				
79		Well Installation				
80		Office of State Engineer Permit Application				
81		Kirtland Dig Permit				
82		Well Installation				
83		IDW Sampling and Letters				
84		OSE Report-Installation				
85		Well Survey				
86		Well Abandonment				
87		Office of State Engineer Permit Application				
88		Well Abandonment				
89		OSE Report-Abandonment				
90		Reporting				
91		Monthly Drinking Water Sampling, Analysis and Reporting CY 2016	271 days	Mon 2/1/16	Mon 2/13/17	
92		Month 1 (Feb 2016)	58 days	Mon 2/1/16	Wed 4/20/16	



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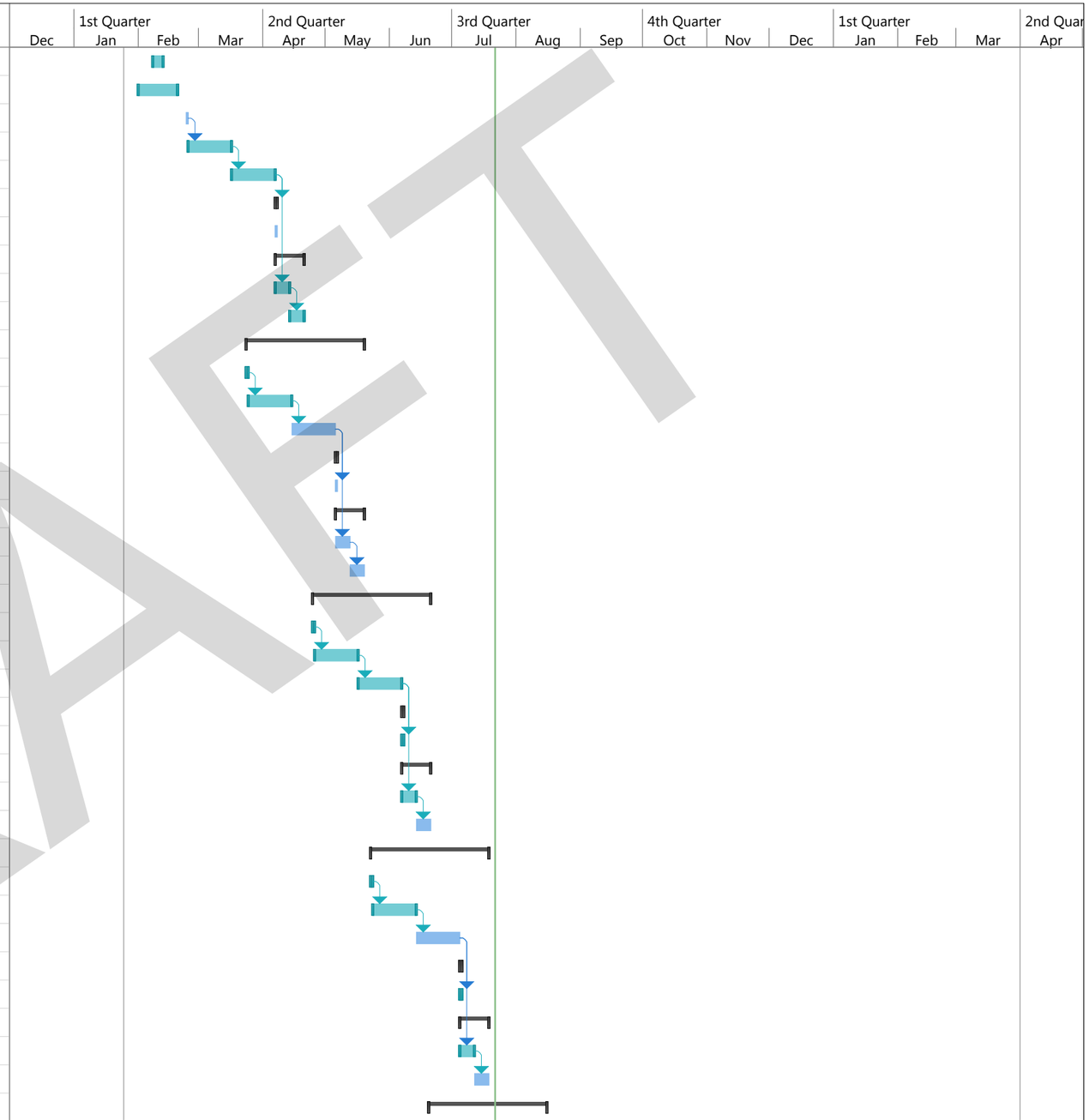
Split	Inactive Task	Duration-only	Finish-only	Progress	
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

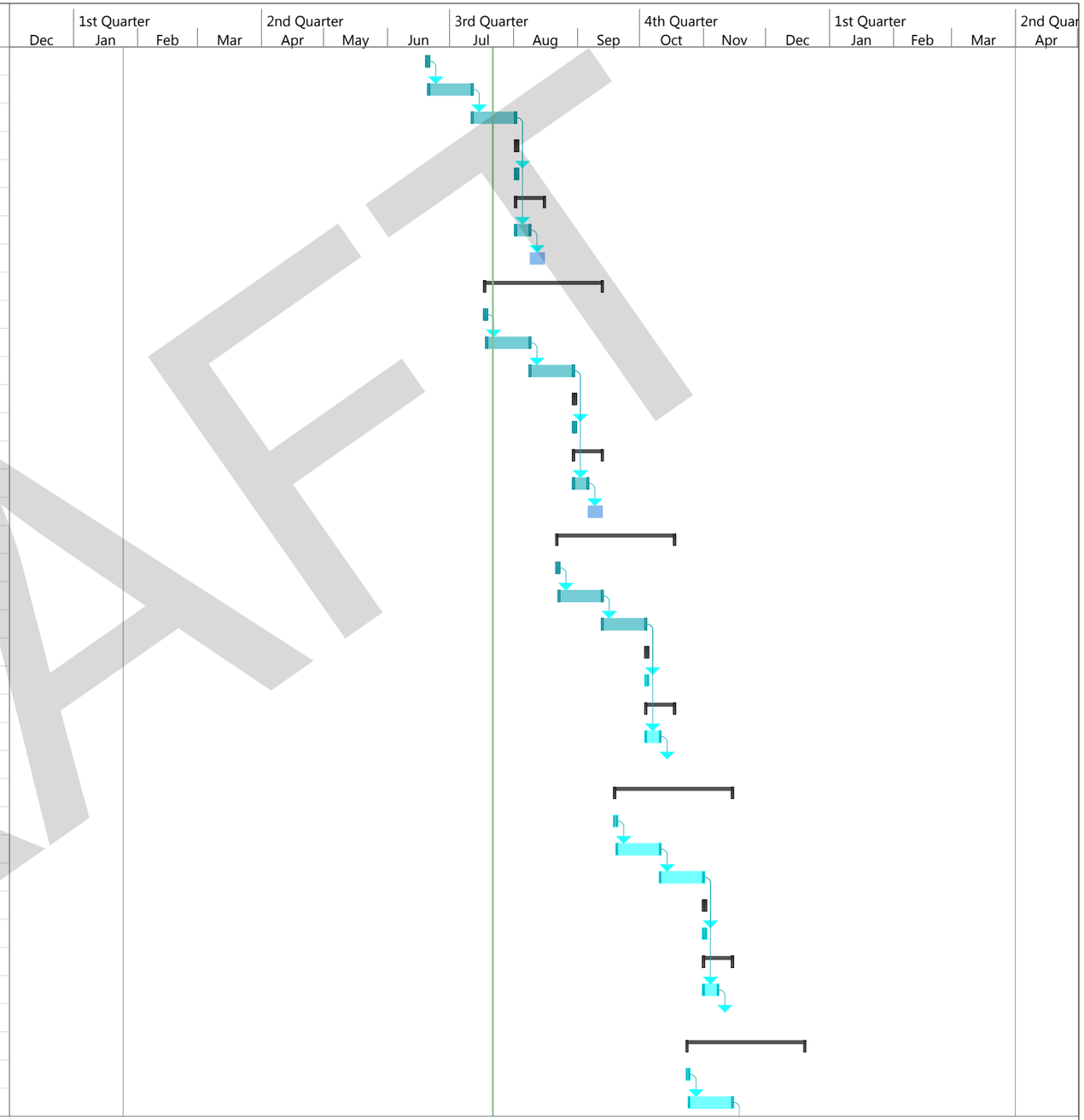
ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
93		Mobilization	5 days	Mon 2/8/16	Fri 2/12/16	
94		Sample Planning	15 days	Mon 2/1/16	Fri 2/19/16	
95		Field Work	1 day	Wed 2/24/16	Wed 2/24/16	
96		Laboratory Analysis	15 days	Thu 2/25/16	Wed 3/16/16	95
97		Data Validation	15 days	Thu 3/17/16	Wed 4/6/16	96
98		Database Management	1 day	Thu 4/7/16	Thu 4/7/16	97
99		ERPIMS uploads	1 day	Thu 4/7/16	Thu 4/7/16	
100		Technical Memo	10 days	Thu 4/7/16	Wed 4/20/16	
101		Draft Technical Memo	5 days	Thu 4/7/16	Wed 4/13/16	97
102		Submit To USACE	5 days	Thu 4/14/16	Wed 4/20/16	101
103		Month 2 (March 2016)	41 days	Thu 3/24/16	Thu 5/19/16	
104		Field Work	1 day	Thu 3/24/16	Thu 3/24/16	
105		Laboratory Analysis	15 days	Fri 3/25/16	Thu 4/14/16	104
106		Data Validation	15 days	Fri 4/15/16	Thu 5/5/16	105
107		Database Management	1 day	Fri 5/6/16	Fri 5/6/16	
108		ERPIMS uploads	1 day	Fri 5/6/16	Fri 5/6/16	106
109		Technical Memo	10 days	Fri 5/6/16	Thu 5/19/16	
110		Draft Technical Memo	5 days	Fri 5/6/16	Thu 5/12/16	106
111		USACE/AFCEC/KAFB Review	5 days	Fri 5/13/16	Thu 5/19/16	110
112		Month 3 (April 2016)	41 days	Mon 4/25/16	Mon 6/20/16	
113		Field Work	1 day	Mon 4/25/16	Mon 4/25/16	
114		Laboratory Analysis	15 days	Tue 4/26/16	Mon 5/16/16	113
115		Data Validation	15 days	Tue 5/17/16	Mon 6/6/16	114
116		Database Management	1 day	Tue 6/7/16	Tue 6/7/16	
117		ERPIMS uploads	1 day	Tue 6/7/16	Tue 6/7/16	115
118		Technical Memo	10 days	Tue 6/7/16	Mon 6/20/16	
119		Draft Technical Memo	5 days	Tue 6/7/16	Mon 6/13/16	115
120		USACE/AFCEC/KAFB Review	5 days	Tue 6/14/16	Mon 6/20/16	119
121		Month 4 (May 2016)	41 days	Mon 5/23/16	Mon 7/18/16	
122		Field Work	1 day	Mon 5/23/16	Mon 5/23/16	
123		Laboratory Analysis	15 days	Tue 5/24/16	Mon 6/13/16	122
124		Data Validation	15 days	Tue 6/14/16	Mon 7/4/16	123
125		Database Management	1 day	Tue 7/5/16	Tue 7/5/16	
126		ERPIMS uploads	1 day	Tue 7/5/16	Tue 7/5/16	124
127		Technical Memo	10 days	Tue 7/5/16	Mon 7/18/16	
128		Draft Technical Memo	5 days	Tue 7/5/16	Mon 7/11/16	124
129		USACE/AFCEC/KAFB Review	5 days	Tue 7/12/16	Mon 7/18/16	128
130		Month 5 (June 2016)	41 days	Mon 6/20/16	Mon 8/15/16	



Project: KAFB_Schedule_072216
Date: Fri 7/22/16

Task	Project Summary	Manual Task	Start-only	Deadline	
Split	Inactive Task	Duration-only	Finish-only	Progress	
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
131		Field Work	1 day	Mon 6/20/16	Mon 6/20/16	
132		Laboratory Analysis	15 days	Tue 6/21/16	Mon 7/11/16	131
133		Data Validation	15 days	Tue 7/12/16	Mon 8/1/16	132
134		Database Management	1 day	Tue 8/2/16	Tue 8/2/16	
135		ERPIMS uploads	1 day	Tue 8/2/16	Tue 8/2/16	133
136		Technical Memo	10 days	Tue 8/2/16	Mon 8/15/16	
137		Draft Technical Memo	5 days	Tue 8/2/16	Mon 8/8/16	133
138		USACE/AFCEC/KAFB Review	5 days	Tue 8/9/16	Mon 8/15/16	137
139		Month 6 (July 2016)	41 days	Mon 7/18/16	Mon 9/12/16	
140		Field Work	1 day	Mon 7/18/16	Mon 7/18/16	
141		Laboratory Analysis	15 days	Tue 7/19/16	Mon 8/8/16	140
142		Data Validation	15 days	Tue 8/9/16	Mon 8/29/16	141
143		Database Management	1 day	Tue 8/30/16	Tue 8/30/16	
144		ERPIMS uploads	1 day	Tue 8/30/16	Tue 8/30/16	142
145		Technical Memo	10 days	Tue 8/30/16	Mon 9/12/16	
146		Draft Technical Memo	5 days	Tue 8/30/16	Mon 9/5/16	142
147		USACE/AFCEC/KAFB Review	5 days	Tue 9/6/16	Mon 9/12/16	146
148		Month 7 (August 2016)	41 days	Mon 8/22/16	Mon 10/17/16	
149		Field Work	1 day	Mon 8/22/16	Mon 8/22/16	
150		Laboratory Analysis	15 days	Tue 8/23/16	Mon 9/12/16	149
151		Data Validation	15 days	Tue 9/13/16	Mon 10/3/16	150
152		Database Management	1 day	Tue 10/4/16	Tue 10/4/16	
153		ERPIMS uploads	1 day	Tue 10/4/16	Tue 10/4/16	151
154		Technical Memo	10 days	Tue 10/4/16	Mon 10/17/16	
155		Draft Technical Memo	5 days	Tue 10/4/16	Mon 10/10/16	151
156		USACE/AFCEC/KAFB Review	5 days	Tue 10/11/16	Mon 10/17/16	155
157		Month 8 (September 2016)	41 days	Mon 9/19/16	Mon 11/14/16	
158		Field Work	1 day	Mon 9/19/16	Mon 9/19/16	
159		Laboratory Analysis	15 days	Tue 9/20/16	Mon 10/10/16	158
160		Data Validation	15 days	Tue 10/11/16	Mon 10/31/16	159
161		Database Management	1 day	Tue 11/1/16	Tue 11/1/16	
162		ERPIMS uploads	1 day	Tue 11/1/16	Tue 11/1/16	160
163		Technical Memo	10 days	Tue 11/1/16	Mon 11/14/16	
164		Draft Technical Memo	5 days	Tue 11/1/16	Mon 11/7/16	160
165		USACE/AFCEC/KAFB Review	5 days	Tue 11/8/16	Mon 11/14/16	164
166		Month 9 (October 2016)	41 days	Mon 10/24/16	Mon 12/19/16	
167		Field Work	1 day	Mon 10/24/16	Mon 10/24/16	
168		Laboratory Analysis	15 days	Tue 10/25/16	Mon 11/14/16	167



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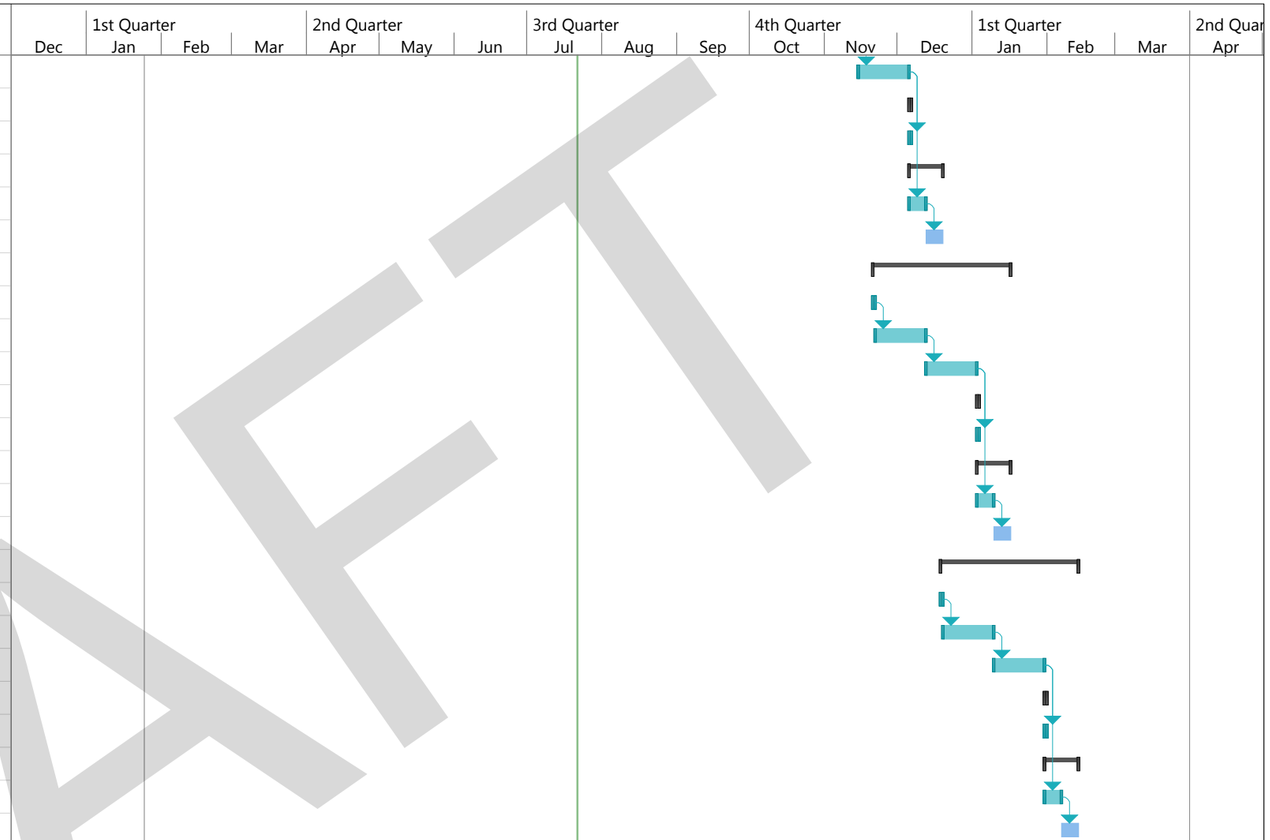
Split	Inactive Task	Duration-only	Finish-only	Progress	
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
169		Data Validation	15 days	Tue 11/15/16	Mon 12/5/16	168
170		Database Management	1 day	Tue 12/6/16	Tue 12/6/16	
171		ERPIMS uploads	1 day	Tue 12/6/16	Tue 12/6/16	169
172		Technical Memo	10 days	Tue 12/6/16	Mon 12/19/16	
173		Draft Technical Memo	5 days	Tue 12/6/16	Mon 12/12/16	169
174		USACE/AFCEC/KAFB Review	5 days	Tue 12/13/16	Mon 12/19/16	173
175		Month 10 (November 2016)	41 days	Mon 11/21/16	Mon 1/16/17	
176		Field Work	1 day	Mon 11/21/16	Mon 11/21/16	
177		Laboratory Analysis	15 days	Tue 11/22/16	Mon 12/12/16	176
178		Data Validation	15 days	Tue 12/13/16	Mon 1/2/17	177
179		Database Management	1 day	Tue 1/3/17	Tue 1/3/17	
180		ERPIMS uploads	1 day	Tue 1/3/17	Tue 1/3/17	178
181		Technical Memo	10 days	Tue 1/3/17	Mon 1/16/17	
182		Draft Technical Memo	5 days	Tue 1/3/17	Mon 1/9/17	178
183		USACE/AFCEC/KAFB Review	5 days	Tue 1/10/17	Mon 1/16/17	182
184		Month 11 (December 2016)	41 days	Mon 12/19/16	Mon 2/13/17	
185		Field Work	1 day	Mon 12/19/16	Mon 12/19/16	
186		Laboratory Analysis	15 days	Tue 12/20/16	Mon 1/9/17	185
187		Data Validation	15 days	Tue 1/10/17	Mon 1/30/17	186
188		Database Management	1 day	Tue 1/31/17	Tue 1/31/17	
189		ERPIMS uploads	1 day	Tue 1/31/17	Tue 1/31/17	187
190		Technical Memo	10 days	Tue 1/31/17	Mon 2/13/17	
191		Draft Technical Memo	5 days	Tue 1/31/17	Mon 2/6/17	187
192		USACE/AFCEC/KAFB Review	5 days	Tue 2/7/17	Mon 2/13/17	191
193		Monthly Drinking Water Sampling, Analysis and Reporting CY 2017				
194		Will follow same schedule as 2016 events				
195		Vadose Zone Sampling, Analysis and Reporting First Quarter CY 2017				
196		Will follow similar schedule to 2016 events				



Project: KAFB_Schedule_072216
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Summary	Inactive Summary	Manual Summary	External Milestone	

Appendix C
Quality Assurance Project Plan

KIRTLAND AIR FORCE BASE
ALBUQUERQUE, NEW MEXICO

Quality Assurance Project Plan for Soil Vapor Monitoring and Drinking Water Monitoring Bulk Fuels Facility Solid Waste Management Unit ST-106/SS-111 Kirtland Air Force Base, New Mexico

August 2016

Prepared for

U.S. Army Corps of Engineers
Albuquerque District
4101 Jefferson Plaza NE
Albuquerque, NM 87109

Contract No. W912PP-16-C-0002

Prepared by

Sundance Consulting, Inc.
8210 Louisiana Blvd. NE Suite C
Albuquerque, NM 87113

DISTRIBUTION LIST
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Ryan Wortman	Sundance Field Team Lead/Deputy SSHO	Sundance	505-835-7660 x155	rwortman@sundance-inc.net

DISTRIBUTION LIST
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded)

Acronyms and Abbreviations:
AFCEC = Air Force Civil Engineer Center
BFF = Bulk Fuels Facility
COR = Contracting Officer's Representative
KAFB = Kirtland Air Force Base
NMED = New Mexico Environment Department
P.E. = Professional Engineer
P.G. = Professional Geologist
PhD = Doctor of Philosophy
PM = Project Manager
POC = point of contact
QAPjP = Quality Assurance Project Plan
SSHO = Site Safety and Health Officer
Sundance = Sundance Consulting, Inc.
USACE = U.S. Army Corps of Engineers

CONTENTS

CONTENTS (CONCLUDED)

ATTACHMENTS

FIGURES

TABLES

ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
%	percent
AFB	Air Force Base
ALS	ALS Environmental Laboratory
APH	air-phase petroleum hydrocarbon
APP	Accident Prevention Plan
ASTM	ASTM International
BFF	Bulk Fuels Facility
bgs	below ground surface
BTEX	benzene, toluene, ethylbenzene, and xylenes
CARB	California Air Resources Board
CO	carbon monoxide
COA	City of Albuquerque
CoC	contaminants of concern
COC	chain-of-custody
COPC	contaminant of potential concern
CY	calendar year
DoD	U.S. Department of Defense
DQA	data quality assessment
DQO	data quality objective
EDB	ethylene dibromide
e.g.	example given
ELAP	Environmental Laboratory Accreditation Program
EPA	U.S. Environmental Protection Agency
ft	foot/feet
HC	hydrocarbon
ID	identification
IDW	investigation-derived waste
KAFB	Kirtland Air Force Base
L	liter
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDC	Laboratory Data Consultants, Inc.
LOQ	limit of quantitation
MA APH	Massachusetts Air-Phase Petroleum Hydrocarbons
MA DEP	Massachusetts Department of Environmental Protection

ACRONYMS AND ABBREVIATIONS (CONTINUED)

MCL	maximum contaminant level
MDL	method detection limit
mL	milliliter
MS	matrix spike
MSD	matrix spike duplicate
NAPL	non-aqueous phase liquid
NCR	Nonconformance Report
NIST	National Institute of Standards and Technology
NMED	New Mexico Environment Department
NMWQCC	New Mexico Water Quality Control Commission
No.	number
NOD	Notice of Deficiency
O_2	oxygen (molecular)
OSRTI	Office of Superfund Remediation and Technology Innovation
OSWER	Office of Solid Waste and Emergency Response
PARCC	precision, accuracy, representiveness, comparability and completeness
P.E.	Professional Engineer
P.G.	Professional Geologist
PM	Project Manager
POC	point of contact
PPE	personal protective equipment
QA	quality assurance
QAPjP	Quality Assurance Project Plan
QC	quality control
QCM	Quality Control Manager
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
RPD	relative percent difference
RSL	regional screening level
Sundance	Sundance Consulting, Inc.
SOP	standard operating procedure
SVE	soil vapor extraction
SVM	soil vapor monitoring
SVMP	soil vapor monitoring point
SVOC	semi volatile organic compound
SWMU	solid waste management unit
TPH	total petroleum hydrocarbons
U.S.	United States
USACE	U.S. Army Corps of Engineers
USAF	U.S. Air Force

ACRONYMS AND ABBREVIATIONS (CONCLUDED)

VA	Veteran's Administration
VOC	volatile organic compound
VPH	volatile petroleum hydrocarbon
WP	Work Plan

SIGNATURE SHEET

SOIL VAPOR MONITORING AND DRINKING WATER MONITORING BULK FUELS FACILITY, SOLID WASTE MANAGEMENT UNIT ST-106/SS-111 KIRTLAND AIR FORCE BASE, NEW MEXICO

August 12, 2016

Ryan Wortman,
Field Team Lead/ Deputy Site Safety and Health Officer
Sundance Consulting, Inc.

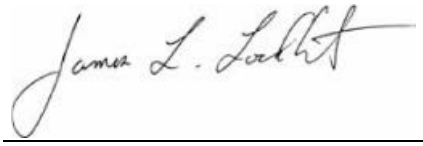
Date

August 12, 2016

Rachel Hobbs, P.G.
Project Manager
Sundance Consulting, Inc.

Date

CERTIFICATION



August 12, 2016

James L. Lockhart, P.E.
Vice President of Operations
Sundance Consulting Inc.

Date

EXECUTIVE SUMMARY

This Quality Assurance Project Plan (QAPjP) has been prepared by Sundance Consulting, Inc. (Sundance) under the U.S. Army Corps of Engineers (USACE)–Albuquerque District, Contract Number W912PP-16-C-0002. This QAPjP was developed to support soil vapor monitoring and drinking water monitoring at Kirtland Air Force Base (KAFB). The work to be conducted under this contract will include periodic monitoring of soil vapor and drinking water, as well as the abandonment and installation of soil vapor monitoring points at a location and date to be determined by USACE.

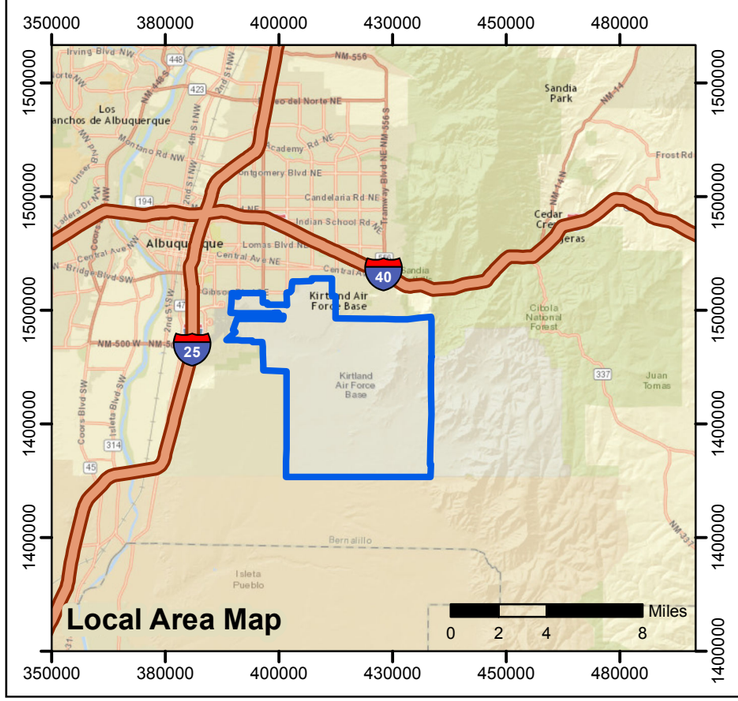
This QAPjP was developed for the periodic monitoring at KAFB Bulk Fuels Facility to meet the quality control requirements defined in the Department of Defense Quality Systems Manual (Version 5.0, July 2013). The QAPjP documents project management procedures and describes data generation and acquisition for field sampling and laboratory analytical processes, laboratory analytical methods, quality assurance/quality control protocols, data validation and usability, assessment and oversight, data management processes, and reporting requirements to be implemented for the project.

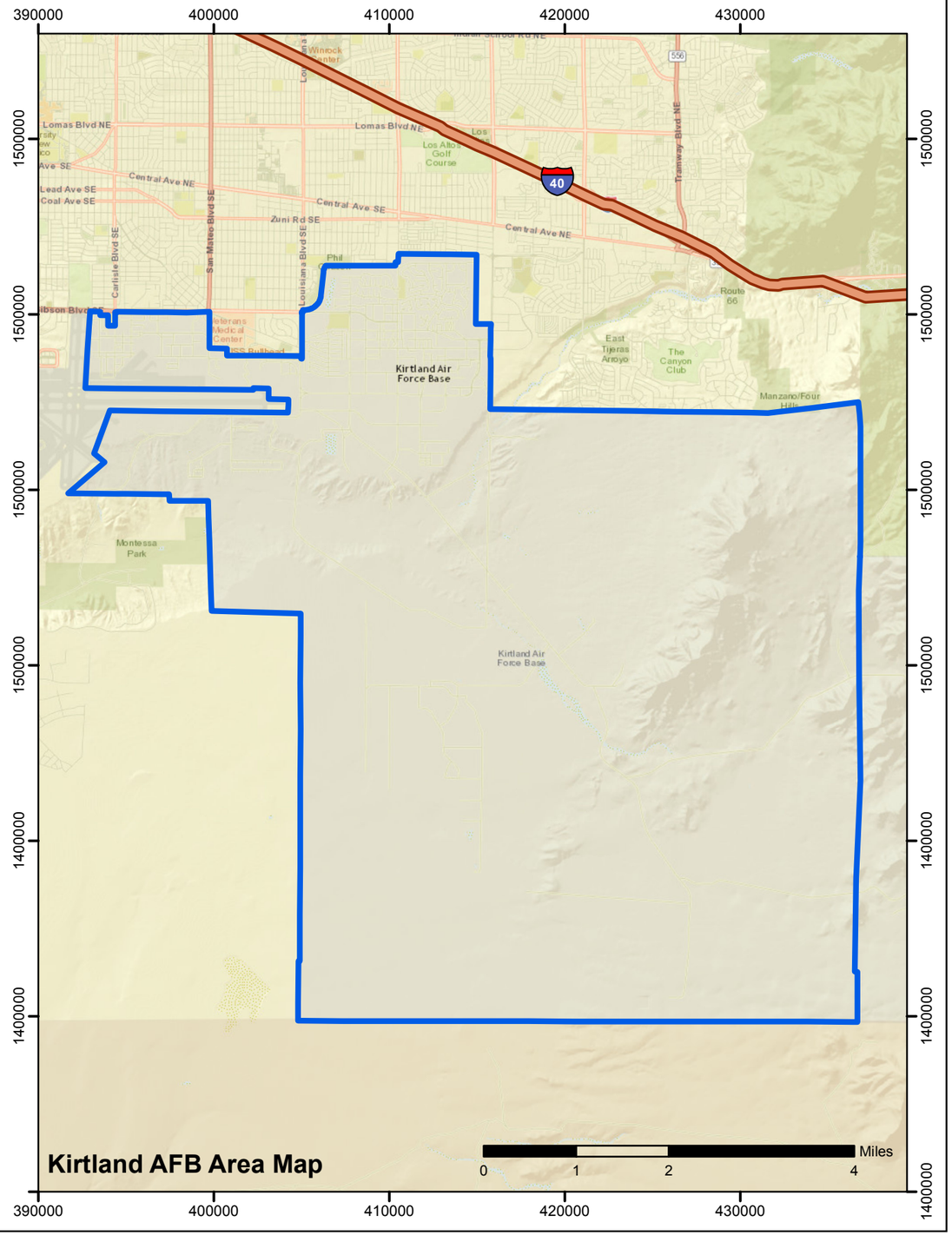
1 INTRODUCTION

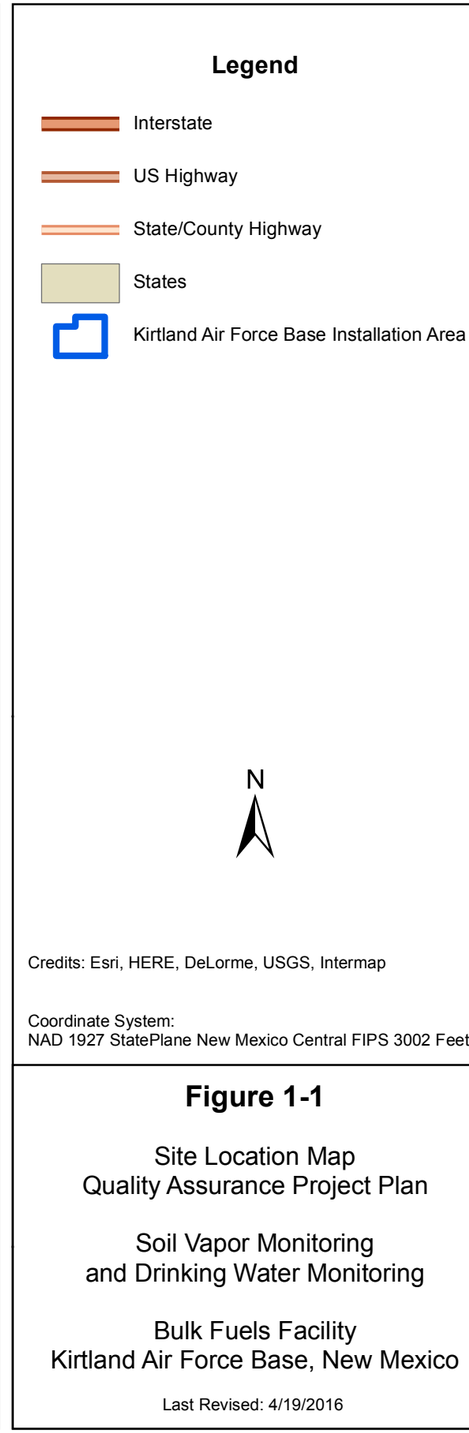
This Soil Vapor Monitoring and Drinking Water Monitoring Quality Assure Project Plan was prepared by Sundance Consulting, Inc. (Sundance) for the United States Army Corps of Engineers (USACE) under contract number W912PP-16-C-0002. Kirtland Air Force Base (KAFB) is located in Bernalillo County, in central New Mexico, southeast of and adjacent to the City of Albuquerque and the Albuquerque International Sunport. The approximate area of the base is 52,287 acres. The Bulk Fuels Facility (BFF) site, comprised of Solid Waste Management Unit (SWMU) ST-106/SS-111, is located in the northwestern part of KAFB (Figure 1-1). Environmental restoration efforts at the BFF site are being conducted under requirements set forth in the Resource Conservation and Recovery Act (RCRA), Permit Number (No.) NM9570024423, with the New Mexico Environment Department (NMED) serving as the lead regulatory agency (NMED 2010). This Quality Assurance Project Plan (QAPjP) addresses activities that are continuing the implementation of the RCRA Interim Measures for the site, including continuation of soil vapor monitoring (SVM) and drinking water supply well monitoring, and the installation and abandonment of one or more soil vapor monitoring points (SVMPs) at locations and dates to be determined by USACE with United States Air Force (USAF) input.

The BFF and associated infrastructure operated from 1953 through 1999. During this time, the fueling area was separated into a tank holding area where bulk shipments of fuel were received, and a fuel loading area where individual fuel railcars or trucks were emptied or disharged. In 1999, KAFB stopped using the underground piping at the facility and removed this piping from service due to discovery of a leak. Although the fuel leak was identified by KAFB, the exact history of the releases is unknown. Releases could have occurred when fuel was transferred from railcars or trucks to the pump house. Initially, it was thought that the leak only affected surface soil around the identified source area; however, KAFB learned through characterization activities that the leaked fuel had migrated to the groundwater table and that dissolved phase fuel contamination had migrated northeast and north of KAFB.

In order to comply with NMED Hazardous Waste Bureau requirements, a RCRA Facility Investigation (RFI) has been ongoing since 2011. As part of this ongoing investigation, 284 SVMPs have been and are sampled to characterize the nature and extent of soil vapor contamination in the vadose zone (approximately 460 feet (ft) from the ground surface to the top of the water table). In addition, drinking water supply wells have been and continue to be monitored to ensure they have not been impacted by contaminants from the BFF site. Under this project, SVM will continue and annual maintenance will be performed at the SVMPs. The drinking water supply well monitoring will continue through sampling and analysis.







Credits: Esri, HERE, DeLorme, USGS, Intermap

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

Figure 1-1

Site Location Map
Quality Assurance Project Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016

2 PROJECT MANAGEMENT AND ORGANIZATION

Project management for the soil vapor and drinking water monitoring activities will be performed in accordance with the requirements and the authority of the USACE, Contract No. W912PP-16-C-0002, and other applicable federal and state regulations.

The BFF project team consists of representatives from USACE, USAF, Sundance and its subcontractors, and the NMED. The USAF is the lead federal agency for direction of site activities and decision-making. The NMED Hazardous Waste Bureau is the lead regulatory agency.

2.1 Project Quality Assurance Organization

The project quality assurance (QA) organization, presented in Figure 2-1, identifies key Sundance individuals and responsibilities to ensure project QA objectives are achieved for soil vapor and drinking water monitoring.

2.2 Personnel Qualifications

Personnel qualifications for key Sundance individuals supporting the monitoring activities are listed in Table 2-1 in addition to the title, responsibility, education, experience, and authority level.

2.3 Task Description

The tasks to be addressed under this QAPjP include quarterly SVM and monthly drinking water supply well monitoring. This document addresses all of the quality aspects of the following tasks:

- Sample SVM network of 284 SVMPs quarterly for volatile organic compounds (VOCs) by method TO-15, ethylene dibromide (EDB) by method California Air Resources Board (CARB) 422, air-phase petroleum hydrocarbons (APH) by Massachusetts Air-Phase Petroleum Hydrocarbon (MA APH) method 1.0, and fixed gases by U.S. Environmental Protection Agency (EPA) method 3C (Figure 2-2).

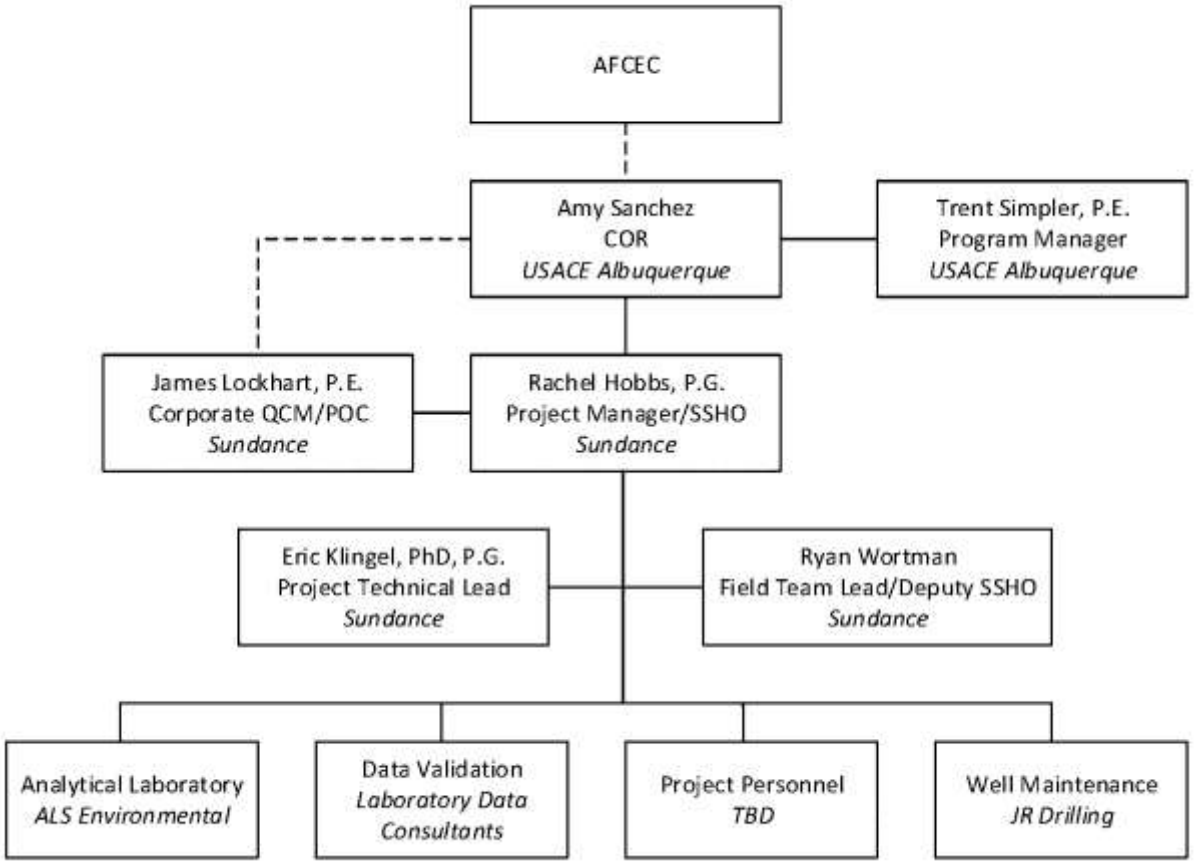
- Sample four drinking water supply wells monthly for EDB by EPA method 504.1, and benzene, toluene, ethylbenzene, and xylenes (BTEX) by EPA Method 524.2 (Figure 2-3).

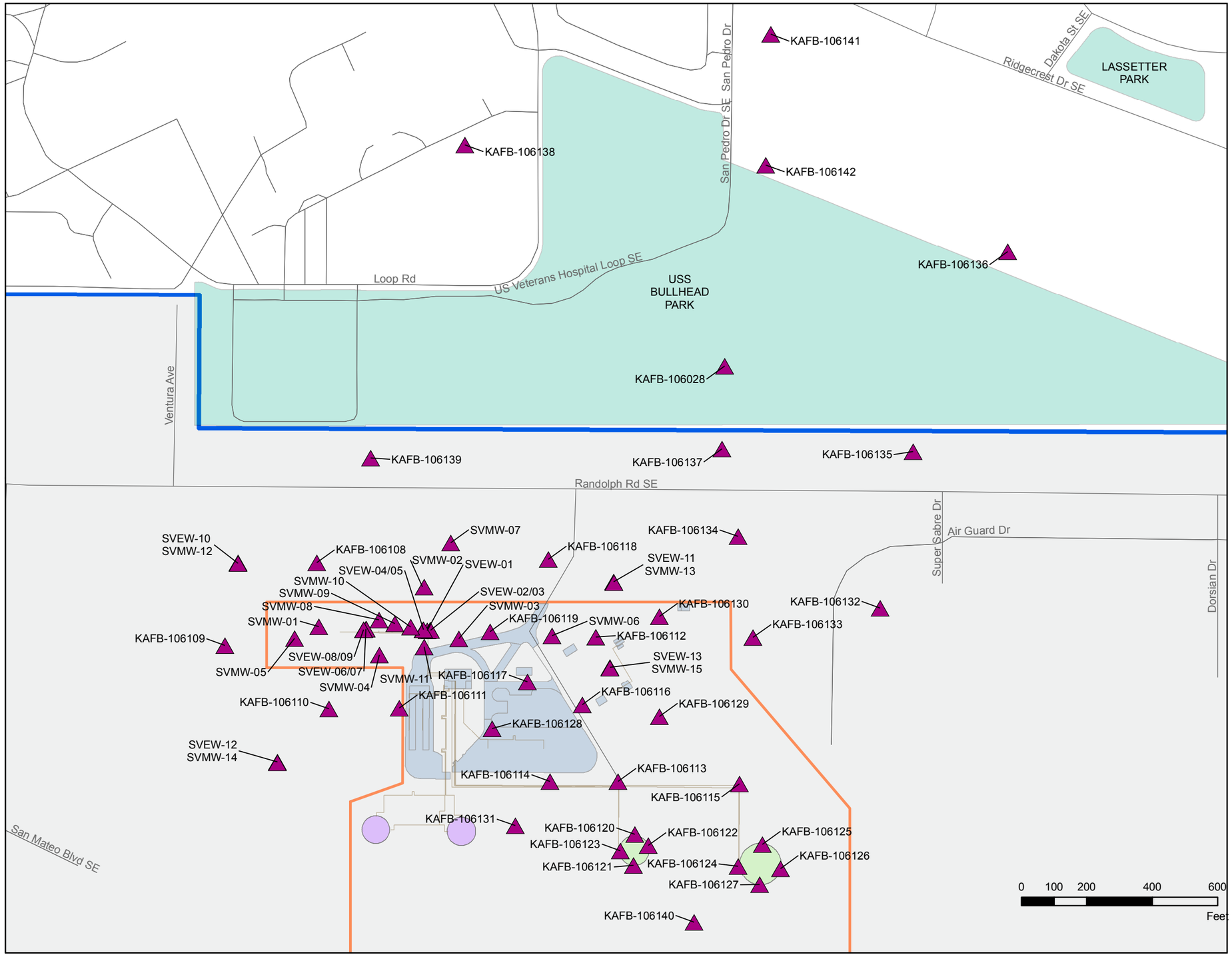
- Perform annual maintenance of the SVM network.

- Abandon and install SVMPs as necessary.

- Analyze soil vapor and drinking water supply well samples, and report results for soil vapor and drinking water supply wells in Quarterly Monitoring and Site Investigation Reports.

Figure 2-1. Quality Assurance Organization
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico





Legend

- Kirtland Air Force Base Installation Area
- City of Albuquerque Parks
- Roads
- Bulk Fuels Facility Area
- Soil Vapor Monitoring Location
- Bulk Fuels Facility Infrastructure
- Current Fuel Storage Tanks
- Former Fuel Storage Tanks
- Fuel Transfer Lines

N

Credits: City of Albuquerque

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

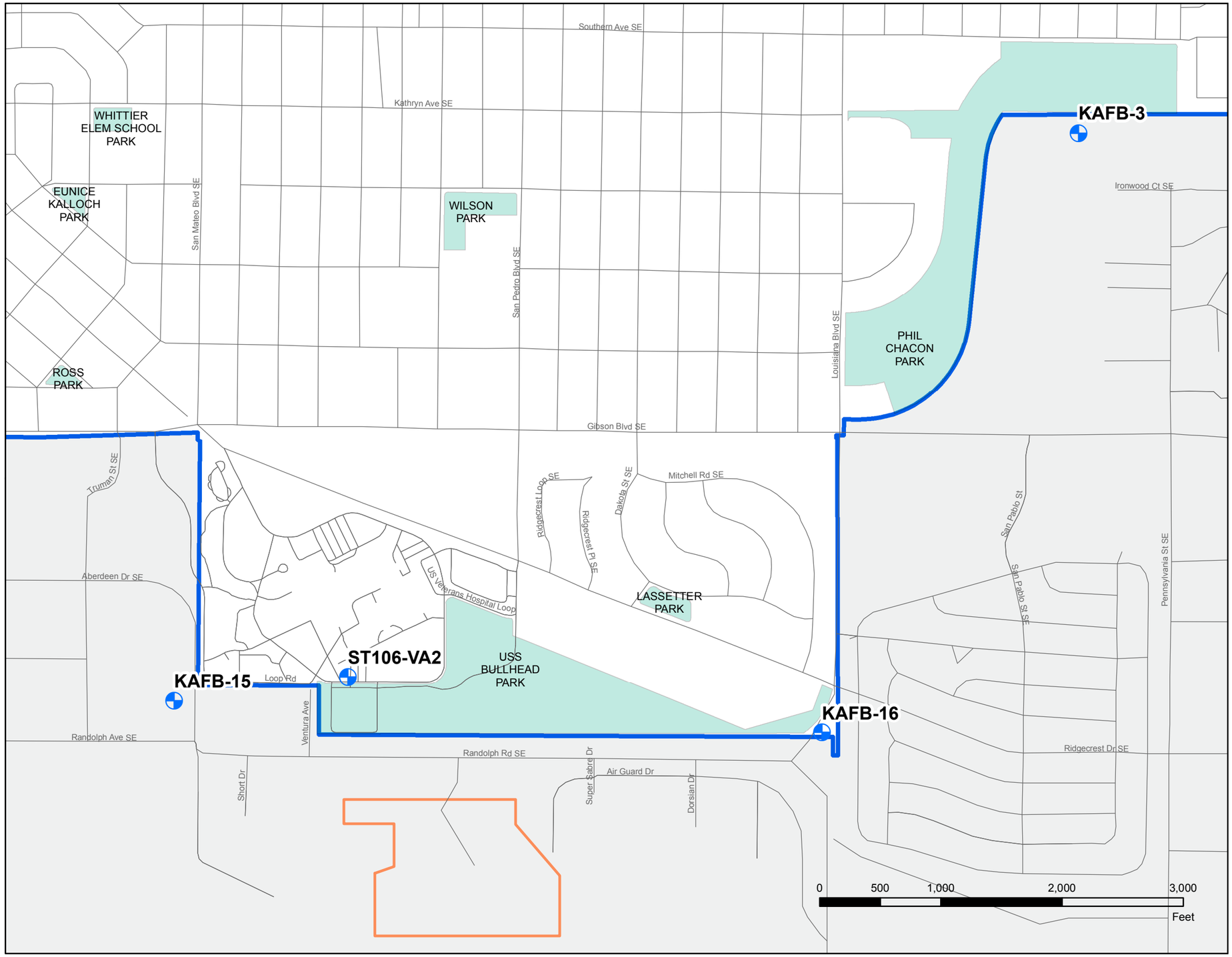
Figure 2-2

Soil Vapor Monitoring Locations
Quality Assurance Project Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016



Legend

- Kirtland Air Force Base Installation Area
- City of Albuquerque Parks
- Roads
- Bulk Fuels Facility Area
- Drinking Water Supply Well

N

Credits: City of Albuquerque

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

Figure 2-3

Drinking Water Supply Wells
Quality Assurance Project Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016

Table 2-1. Personnel Qualifications
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Position/Staff	Qualifications	Responsibilities	Authority Level
Vice President of Operations: Jim Lockhart, P.E.	• BSME, MBA; • 33 years' experience in environmental remediation and engineering; • 25 years in management of environmental and engineering projects.	• As Sundance Officer, authorized to negotiate and commit resources; • Primary POC for USACE on contractual and programmatic items; • Ensures consistency in deliverables and cost/performance reporting and progress reporting/invoicing; • Coordinates issue resolution as needed with the COR and/or CO.	• Coordinates corrective action at programmatic level.
Project Manager/SSHO: Rachel Hobbs, P.G.	• M.S. in Geology; • Registered Professional Geologist in the state of Tennessee; • 5 years' experience in environmental remediation; • Past experience coordinating Kirtland BFF project tasks.	• Reports to the PM and serves as the Alternative PM; • Overall responsibility for design, implementation, and management of sampling activities; • Reviews all WP, reporting, and data deliverables; • Coordinates with Field Personnel for oversight and quality control; • Responsible for providing input for the design of the corrective actions and reviews corrective elements specific to sampling; • Oversees development of APP in accordance with Engineer Manual 385-1-1 and Occupational Safety and Health Administration regulations; • Assists PM and procurement staff in verification of safety performance of Sundance staff and subcontractors. Investigates any incidents, accidents, or safety violations Performs safety audits; • Manages monitoring reports.	• Approves APPs/SSHPs and all modifications before issuance to USACE; • Manages Health and Safety Program and directs training and required attendance; • Investigates safety concerns raised by staff; • Investigates any accidents; • Stops work for any reason including noncompliance/safety violation, or quality violations.

Table 2-1. Personnel Qualifications
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 2 of 3)

Project Technical Lead: Eric Klingel, PhD, P.G.	• PhD. in Geology; • Registered Professional Geologist in the state of North Carolina and South Carolina; • 30 years' experience in environmental site characterization, remediation and project management.	• Reports to the Project Manager and serves as the Alternative Project Manager; • Overall responsibility for design, implementation, and management of sampling activities; • Reviews all work plans, reporting, and data deliverables; • Coordinates with Field Personnel for oversight and QC; • Responsible for providing input for the design of the corrective actions and reviews corrective elements specific to sampling; • Oversees development of APP in accordance with Engineer Manual 385-1-1 and Occupational Safety and Health Administration regulations; • Assists Project Manager and procurement staff in verification of safety performance of subcontractors Investigates any incidents, accidents, or safety violations Performs safety audits; • Manages monitoring reports.	• Stops work for any reason including noncompliance/safety violation, or quality violations.
Field Team Lead/Deputy SSHO: Ryan Wortman	• B.S. in Geology; • 1+ year past experience coordinating Kirtland BFF project tasks.	• Reports to Technical Lead and/or PM; • Oversees sampling team and sampling activities; • Coordinates with the PM and Project Technical Lead on any deviations from the QAPjP due to changed field conditions such that data quality objectives are met; • Coordinates with SSHO to ensure that project activities are being performed in accordance with the APP. • Performs Health and Safety oversite in SSHO's absence.	• Stop sampling work at any time due to safety or quality violations.

Table 2-1. Personnel Qualifications
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 3 of 3)

Acronyms & Abbreviations:
APP – Accident Prevention Plan
BFF – Bulk Fuels Facility
B.S. – Bachelor of Science Degree
BSME – Bachelor of Science in Mechanical Engineering
CO – Contracting Officer
COR – Contracting Officer's Representative
DoD – Department of Defense
MA – Master of Arts
MBA – Master of Business Administration
M.S. – Master of Science Degree
P.E. – Professional Engineer
P.G. – Professional Geologist
PhD – Doctor of Philosophy
PM – Project Manager
POC – point of contact
QAPjP – Quality Assurance Project Plan
SSHO – Site Safety and Health Officer
SSHP – Site Safety and Health Plan
USACE – United States Army Corps of Engineers
USAF – United States Air Force

3 DATA GENERATION AND ACQUISITION

3.1 Sampling Design

This section discusses the sampling and analysis strategy for drinking water and soil vapor samples required to meet the project data quality objectives (DQOs). SVM locations and drinking water supply wells are shown in Figures 2-2 and 2-3. Soil samples may be collected as part of the installation of soil vapor monitoring points. If soil sampling is performed as part of this project, the text, tables, and figures of this QAPjP will be revised to include necessary information.

Drinking water and soil vapor samples will be labeled, packaged, and shipped to the environmental Laboratory. Drinking water samples will be shipped to TestAmerica Laboratories in Savannah, Georgia. Soil vapor samples will be shipped to ALS Environmental Laboratory, Inc. (ALS) in Simi Valley, California. ALS and TestAmerica maintain U.S. Department of Defense (DoD) Environmental Laboratory Accreditation Programs (ELAP) certification for the analyses required under this contract. Laboratory Data Consultants, Inc. (LDC) will perform the third party data validation utilizing established data validation procedures (manually or automated) to perform 100 percent (%) review and EPA Level III data validation.

3.1.1 Soil Vapor Monitoring

There are 56 SVM locations, most of which are installed in nested SVM locations that contain multiple monitoring points for a total of 284 SVMPs (Figure 2-2). Based on information that will be collected throughout the implementation of this project, the network may be modified. New SVMPs may be added and some may be removed from the sampling program based on Vadose Zone Working Group recommendations and agreements with NMED. Soil vapor sampling will be performed in accordance with the Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP), to which this QAPjP is attached (Appendix C).

Soil vapor samples will be analyzed for the following parameters:

- VOCs and total petroleum hydrocarbon (TPH) gasoline: EPA Method TO-15
- EDB: CARB 422
- APH (C5-C8 and C9-C12): Massachusetts Department of Environmental Protection (MA DEP)
- Fixed gases (hydrogen, oxygen [O_2], nitrogen, carbon monoxide, carbon dioxide, methane): E3C

3.1.2 Drinking Water Supply Well Monitoring

The drinking water supply well investigation activities at the BFF covered under this QAPjP are limited to four drinking water supply wells (KAFB-3, KAFB-15, KAFB-16, and ST106-VA2). These existing drinking water supply wells at KAFB and the Veteran's Administration (VA) Hospital area able to actively provide drinking water to the facilities' employees and inhabitants. All operational drinking water supply well locations will be sampled monthly, and analyzed for EDB using EPA Method 504.1, and for BTEX using EPA Method 524. Drinking water well sampling will be performed in accordance with the WP.

3.1.3 Soil Vapor Monitoring Well Installation, and Analysis

As part of the vadose zone sampling and analysis, one or more additional SVM locations, consisting of up to six SVMPs approximately 450 ft below ground surface (bgs), may need to be installed. Depending on the objectives of the borehole drilling and well installation, soil samples may be collected during borehole advancement. Soil sample collection techniques, analytical methods, and criteria for collection will be specified in the associated WP. Soil and subsequent soil vapor samples are collected for chemical analysis and will be shipped to ALS for analysis in accordance with DoD Quality Systems Manual (QSM) Version 5.0 (DoD 2013) and laboratory-specific standard operating procedures (SOPs).

3.1.4 Investigation-Derived Waste Management

It is not anticipated that any soil or water investigation-derived waste (IDW) will be generated during monitoring. SVM does not generate any containerized waste. In addition, any excess drinking water will be disposed of via KAFB's waste water treatment system or to the ground. Non-reusable personal protective equipment (PPE) will be disposed of in accordance with the project Accident Prevention Plan (APP) (Section 9.0). Any additional waste associated with sampling (plastic bags, paper waste, etcetera) will be collected and disposed of via the City of Albuquerque's (COA) waste management system.

IDW management details pertaining to any SVM well installation or abandonment that may be performed will be provided in future revisions of, or addendums to the WP. Once the drilling techniques and waste streams are determined, specifications will be submitted describing the management of that waste.

3.2 Quality Objectives and Criteria for Measurement Data

The DQO process is designed to ensure that the type, quantity, and quality of environmental data used for decision-making is appropriate for the intended application. The DQOs for the data collected in association with soil vapor and drinking water well monitoring includes the following:

- Support ongoing monitoring of the SVM network to evaluate soil vapor contamination in the vadose zone

- Support ongoing monitoring to ensure that dissolved phase EDB and BTEX have not impacted the existing drinking water wells

Soil vapor and drinking water well monitoring was initiated in the first quarter of calendar year (CY) 2016.

3.2.1 Comparison Criteria

Analytical methods selected for the project will provide sufficient sensitivity to meet the DQOs and NMED requirements, and will achieve the respective regulatory standard for all analytes in soil vapor, drinking water, and soil.

There are currently no applicable screening standards for soil vapor contamination used for the SWMU ST-106/SS-111 project.

Analytical results from the drinking water supply well monitoring events will be compared to EPA maximum contaminant levels (MCL) and New Mexico Water Quality Control Commission (NMWQCC) standards contained in New Mexico Administrative Code Title 20 – Environmental Protection, Chapter 6 – Water Quality, Part 2 – Ground and Surface Water Protection Section 20.6.2.3103.

Soil samples associated with SVM well installation or abandonment will be compared to EPA residential regional soil screening levels (RSL) (EPA, 2015) and NMED soil screening levels for residential receptors (NMED, 2015). Currently, there are no established regulatory standards for soil vapor. Regulatory limits are summarized in Attachment A with the laboratory analytical methods reporting limits.

Analytical methods, reporting limits, and screening criteria are presented in Attachment A, Tables A-1 and A-2. Analytical methods used by TestAmerica and ALS and reporting limits will provide sufficient sensitivity to meet the DQOs, EPA MCLs, and NMWQCC standards.

3.2.2 Project Performance and Acceptance Criteria

To limit uncertainty in obtained environmental data, criteria for the sensitivity, precision, bias, representativeness, completeness, and comparability (PARCC) parameters were developed and are presented in this QAPjP. Measurement errors will be controlled by using appropriate sampling and analytical methods, adhering to the DoD QSM (2013), following established SOPs, and having data review to verify laboratory processes. Field crews will be trained in appropriate sample collection procedures and will review the QAPjP before sample collection to limit sample collection errors. Subcontract analytical laboratories will have a copy of the QAPjP and will adhere to DoD QSM guidance to limit measurement errors. Following DoD QSM requirements, laboratories will conduct detection limit studies to verify method sensitivity. In addition, laboratories will perform limit of quantitation (LOQ) studies to verify precision and bias at the LOQ. For each matrix and each method, laboratories will analyze applicable QC samples, including laboratory method blanks, surrogates, laboratory control samples (LCS)/laboratory control sample duplicates (LCSD), matrix spike (MS)/matrix spike duplicates (MSD), and internal standards to determine that results of these QC samples are within acceptable precision and bias limits. Acceptance criteria for precision, bias, and sensitivity are presented in Attachment B. The data that meet these criteria will be of definitive quality and of less uncertainty than data which was acquired with a less rigorous approach.

3.3 Monitoring Methods

Soil vapor and drinking water monitoring will be performed in accordance with sampling methodologies presented in Sections 3.2 and 3.3 of the WP and the KAFB Basewide Plans (USAF, 2004).

3.3.1 Equipment Decontamination Procedure

The objective of field decontamination is to remove contaminants of concerns (CoC) from monitoring, and field equipment to concentrations that will not impact study objectives.

Decontamination procedures specific to SMV are outlined in Section 3.2.1.2 of the WP. It is not anticipated that any additional decontamination procedures will be required for soil vapor monitoring.

Decontamination procedures specific to drinking water supply well sampling are outlined in Section 3.3 Step 2 of th WP. It is not anticipated that any additional decontamination procedures will be required for drinking water supply well sampling.

3.4 Sample Handling and Custody

The following sections describe sample packaging and shipment, sample numbering and labeling, and chain-of-custody (COC) requirements associated with collecting soil vapor and drinking water samples.

3.4.1 Sample Packaging and Shipment

Soil vapor and drinking water samples will be collected in the appropriate certified clean sample containers provided by TestAmerica or ALS, and in accordance with the specific WP procedures and Table 3-1.

The primary objective of sample packaging and shipping requirements is to maintain sample integrity from the time a sample is collected until it is received at the analytical laboratory. Specific procedures for packaging and shipping of environmental samples are presented below:

Step 1. A sample label is attached to the sample bottle and completed with indelible ink;

Step 2. For water samples, a cooler will be used as a shipping container. In preparation for shipping samples, the drain plug will be taped shut so that no fluids, such as melted ice, will drain out of the cooler during shipment. A large plastic bag may be used as a liner for the cooler and packing material, such as bubble wrap, or Styrofoam beads, will be placed in the bottom of the liner. All water samples for chemical analysis must be shipped cooled to 4 degrees Celsius (°C) with ice. All samples will require icing prior to shipment. A temperature blank will be placed in every cooler shipment.

Step 3. Soil vapor samples will be returned to the lab in the soil vapor container boxes in which they were received. There are no temperature or preservative requirements for shipping of soil vapor samples.

Step 4. The soil vapor container liner will be taped closed, if used, and sufficient packing material will be used to prevent sample containers from making contact or rolling around during shipment.

Step 5. A copy of the COC form will be placed inside the sample cooler or box.

Step 6. The sample cooler or box will be closed and taped shut with packing tape.

Step 7. Custody seals will be placed on the sample cooler or box.

Step 8. The sample cooler or box will be shipped in accordance with the particular sample media and corresponding hold times.

3.4.2 Soil Vapor Monitoring Well and Drinking Water Supply Well Field Sample Identification

Field sample identification (ID) will be assigned consistent with the established KAFB sample ID nomenclature for soil vapor monitoring well and drinking water supply well field sample IDs. This will ensure that monitoring data associated with the BFF investigation will be recognizable and easily identified once uploaded to the USAF data repository.

3.4.2.1 Monitoring Well IDs

SVM well IDs will follow the format of the base designator (KAFB), the SWMU identifier (106) and the sequential monitoring well number (XXX). Well numbers will follow sequentially those wells that have already been installed at the BFF site.

3.4.2.2 Field Sample IDs

Sample IDs for soil vapor and drinking water samples will be assigned with a consistent and sequential sample number such that the laboratory will not be able to distinguish between samples of the same events. The designation for field samples will be as follows:

- Soil vapor – VA (last two digits of current year) then XXXX. Soil vapor samples collected in 2016 will be labeled VA16XXXX.
- Drinking water – GW (last two digits of current CY) then XXX. Drinking water supply well samples collected in 2016 will be labeled GW16XXX.

3.4.2.3 Field Quality Control Sample IDs

Field duplicate samples will have designations consistent with the sequential field sample IDs such that they will not be distinguishable by the laboratories as being a duplicate sample. Matrix spike (MS) and matrix spike duplicates (MSD) samples, trip blank, and field blank, samples will have sample designations as listed below:

- MS: GW16XXX-MS or -MSD
- Trip blanks (VOCs): GW16TB01, VA16TB01
- Field blanks/ambient blanks (VOCs): GW16AB01, VA16AB01

3.4.3 Sample Custody and Documentation

Sampling information will be recorded on a COC form and sample collection forms for tracking. All entries will be legible and recorded in indelible ink. Because samples may be analyzed at multiple laboratories, the terms laboratory and sample custodian are generic. The custody procedures described herein apply to all laboratories that are involved in the analysis of soil vapor, drinking water, and soil samples.

3.4.3.1 Chain of Custody Records

A blank example COC form is included in Appendix A of the WP. In addition to providing a custody exchange record for the samples, the COC serves as a formal request for sample analyses. The COC form will be completed and signed, thus becoming the COC record and distributed as follows:

- One copy retained by the sample coordinator for inclusion in the project files
- The original sent to the analytical laboratory with the sample shipment

After the laboratory receives field samples, the sample custodian will inventory each shipment before signing for it, and note on the original COC record any discrepancy in the number of samples, temperature of the cooler, or presence of broken samples. The Sundance PM and/or technical lead will be notified immediately of any problems identified with shipped samples, and will determine the appropriate course of action and if project budget or schedule may be impacted.

The laboratory will initiate an internal COC that will track the sample within the various areas of the laboratory. The relinquishing signature of the sample custodian and the custody acceptance signature of the laboratory personnel document that custody of the sample has been transferred appropriately. This procedure will be followed each time a sample changes hands. The laboratory will archive the samples

and maintain them in custody as required by the contract or until further notification from Sundance, at which time the samples will either be returned to the project for disposal, or disposed of by the laboratory.

3.4.3.2 Field Sample Custody

The COC form or record will be the controlling document to ensure that sample custody is maintained. Upon collecting a sample, sampling personnel will initiate the COC in the field. Each individual who has the sample(s) in their possession will sign the COC. Each time the sample custody is transferred, the former custodian will sign the COC on the "Relinquished by" line, and the new custodian will sign the COC on the "Received by" line. The date, time, and name of their project or company affiliation will accompany each signature.

The waybill number or courier name will be recorded on the COC form when a commercial carrier is used. The shipping container will be secured with two custody seals, thereby allowing shipping personnel to maintain custody until receipt by the laboratory.

If the laboratory sample custodian judges sample custody to be invalid (e.g., custody seals have been broken), the laboratory will notify the Sundance PM and/or technical lead who will in turn contact the field team to resolve any discrepancies with field sample documentation. Any corrections required to be made to COC forms will be made by the field team, reviewed by the Sundance PM and/or technical lead to determine impact to sample custody, and transferred to the laboratory. Sample receipt discrepancies will be noted by the laboratory upon sample login.

3.4.3.3 Sample Collection Log

The Sample Collection Log form will be used to document all samples collected in the field. A copy of this form for soil vapor and drinking water can be found in Appendix A of the WP. All entries will be recorded in indelible ink. The sample team will cross out any unused portions and sign each page.

3.4.3.4 Vapor Purge Log

The Vapor Purge Log form will be used to document field sample collection information associated with SVM. A copy of this form can be found in Appendix A of the WP. All entries will be recorded in indelible ink, and will be reviewed by the sampling team. At a minimum, the vapor purge log will contain the following information:

- Project name and site
- SVMP identification number
- Field team /personnel name
- Sample date and time
- Weather conditions
- SVMP observations
- Purge calculations
- Purge volume

- Field measurements (carbon dioxide, O_2, and hydrocarbons [HC])

The Vapor Purge Log will undergo an independent QC review by a field team member other than the author or designee before shipping the samples to the offsite laboratory.

3.4.3.5 Document Corrections

Changes or corrections to any project, field, or analytical documentation will be made by crossing out the item with a single line, initialing by the person performing the correction, and dating the correction. The original item, although erroneous, will remain legible beneath the cross out. The new information will be written above the crossed-out item. Corrections will be written clearly and legibly with indelible ink.

3.5 Analytical Methods

Analytical methods, container, and preservative requirements for soil vapor and drinking water samples are summarized in Tables 3-2 and 3-3. The required target analytes for each method, applicable regulatory limits, project reporting limits, and laboratory LOQs are presented in Attachments A and B.

3.6 Quality Control

This section discusses field and laboratory QC requirements.

3.6.1 Field Quality Control Samples

Field QC samples will be collected and analyzed during the project to assess the precision and accuracy of the sampling program. Field QC samples for this project will include MS/MSD samples, field duplicates, trip blanks for VOC samples, and temperature blanks, and QA split samples if requested by USACE and NMED as discussed below.

3.6.1.1 Matrix Spike and Matrix-Spike Duplicate

MS/MSD samples will be collected at one pair per 20 drinking water samples; at least one per sampling event. MS/MSD analyses will not be performed on soil vapor samples as MS/MSD analysis for these methods and matrix are not applicable. Accuracy for these analyses will be assessed through a review of field duplicates, laboratory duplicates, and surrogate recoveries (when applicable). Field personnel will collect extra volumes for water for MS/MSD analysis and designate the MS/MSD sample(s) on the COC record (Appendix A of the WP).

3.6.1.2 Field Duplicates

Field duplicate pairs consist of two samples of the same matrix (a primary and a duplicate) collected at the same time and location to the extent possible, using the same sampling techniques. The purpose of field duplicate samples is to evaluate sampling precision. Field duplicate samples will be collected for soil vapor monitoring and drinking water sampling. Field duplicate samples will be collected at a frequency of 10% and will be analyzed for the same analytical parameters as their corresponding primary samples. For this project, the acceptance criteria for field duplicate precision is established at less than or equal to 35% for drinking water samples, and 50% for soil vapor samples. Field duplicate precision will be calculated when target analytes are detected above the reporting limit in both the primary and duplicate sample.

No field duplicates will be collected for IDW characterization purposes.

3.6.1.3 Performance Evaluation Samples

Use and anaylsis of performance evaluation samples will be implemented by the client or designee if deemed necessary. Performance evaluation samples are independent clean matrix samples that are spiked with project-specific target compounds and introduced into the sampling program by the field team.

Performance evaluation samples are then submitted to the project laboratory for analysis as blind samples to be evaluated by the USAF upon receipt of data deliverables. These results may serve as an independent QA check for the field sampling and analytical method protocol precision.

3.6.1.4 Trip Blanks

Trip blank samples will accompany each shipment containing soil vapor, drinking water and soil samples for VOC analysis. Trip blanks for drinking water samples will be 40-milliliter (mL) volatile organic analysis vials that contain analyte-free water, which are kept with the field samples during sampling and shipment to an offsite laboratory. Trip blanks for soil vapor samples are 1-liter Bottle-Vacs that are kept with field samples during soil vapor sampling and shipment to an offsite laboratory. The vacuum of the Bottle-Vac will be recorded, but the valve will not be opened, and the container will be returned to the lab with the shipment of soil vapor samples. Results of trip blank samples will be used to determine if samples have been contaminated with VOCs during sampling or shipment to the laboratory.

3.6.1.5 Temperature Blanks

Each cooler containing drinking water samples will be shipped with a temperature blank. A temperature blank is a sample container filled with tap water and shipped in the cooler to the offsite laboratory. The laboratory will record the temperature of the blank upon receipt of the samples. The temperature blank is to ensure that the temperature of the samples when received at the laboratory is less than or equal to 4°C. Temperature blanks are not required to accompany soil vapor samples to the offsite laboratory.

3.6.2 Laboratory Quality Control Samples

To ensure acceptable data quality, laboratory QC analysis will be performed for each method and for each matrix. Laboratory QC samples will include method blanks, initial and continuing calibration blanks, surrogates, LCSs, and internal standards. Tables 3-4 and 3-5 present these QC samples, acceptance criteria, and corrective actions. These QC requirements are consistent with the DoD QSM (2013) guidance. The DoD QSM and laboratory in-house control limits are presented in Attachment B.

3.7 Instrument/Equipment Testing, Inspection, and Maintenance

Field and analytical instrument testing, inspection, and maintenance requirements are described in this section. All requirements are presented in tabular format on Table 3-6 (Field Instrument Quality Control), Table 3-7 (Laboratory Instrument Quality Control – Drinking Water Monitoring), and Table 3-8 (Laboratory Instrument Quality Control – Soil Vapor Monitoring), and in Attachment B.

Other activities such as well installation and abandonment may be performed as part of this project. When designs for these activities are finalized, any additional field measurement specifications for soil boring logs, well reports, and surveying will be included in a subsequent revision to this QAPjP.

3.8 Laboratory Instrument/Equipment Calibration and Frequency

Laboratory instrument calibration requirements, frequencies, and corrective actions for each method in this section. These calibration requirements are established in accordance with the DoD QSM requirements. Calibration is a reproducible reference point to which all sample measurements can be

correlated. Instrumentation calibration is necessary for accurate sample quantitation. Calibrations establish the dynamic range of an instrument, establish response factors to be used for quantitation, and demonstrate instrument sensitivity.

All calibration requirements are presented in tabular format in Table 3-9 (Analytical Instrument Calibration - Gas Chromatography/Mass Spectrometry), and Table 3-10 (Analytical Instrument Calibration (Gas Chromatography), and Attachment B.

3.9 Inspection/Acceptance of Supplies and Consumables

The accuracy of sample target analyte quantitation is directly related to the accuracy of the standards used for instrument calibration. To ensure the highest quality standard, primary reference standards used by laboratories are obtained from reliable commercial sources. Inorganic standards must be traceable to the National Institute of Standards and Technology (NIST); organic standards must be traceable to NIST or American Association of Laboratory Accreditation vendors when available. When standards are received at the laboratory, the date received, supplier, lot number, purity and concentration, and expiration date are recorded in a standard preparation log book. Vendor certifications sent with the standards are also filed and are available upon request.

Standards purchased by the laboratory may be in a pure form, in a stock, or in a working standard solution. All standards made are given a standard identification number and have the following information recorded in a standards log book: source of standard used to prepare dilution; preparer's initials; initial concentration; final concentration; solvent; source and lot number of solvent; volume of final solution; and volume of standard diluted. Records must unambiguously trace the preparation of standards, their use in calibration, and the quantitation of sample results. After preparation and before routine use, the identity and concentration of standards are verified. Verification procedures include a check for chromatographic purity (if applicable) and verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source. Reagents are also examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used. Standards are routinely checked for signs of deterioration (e.g., discoloration, formation of precipitates, or changes in concentration) and are discarded if deterioration is suspected or their expiration date has passed. Expiration dates may be taken from the vendor recommendation, the analytical methods, or from internal research.

Table 3-1. Sample Requirements for Analytical Testing
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Drinking Water and Soil Vapor Samples					
Matrix	**Parameter[1]**	**Container[2,3]**	**Preservation**	**Maximum Holding Times[4]**	
				Extraction	**Analysis**
Water	BTEX	3 x 40-mL G, Septa Vial	Ice to 4°C 4 drops conc. HCl to pH<2	---	14 days
Water	EDB	3 x 40-mL G, Septa Vial	Ice to 4°C	---	14 days
Vapor	VOCs/APH	1 x 1-L Bottle Vac	None	N/A	28 days
Vapor	Fixed gases	1 x 1-L Bottle Vac	None	N/A	30 days
Vapor	CARB 422	1 x 1-L Bottle Vac	None	N/A	30 days

Acronyms and Abbreviations:
< = less than
°C = degrees Celsius; APH = air-phase petroleum hydrocarbon
BTEX = benzene, toluene, ethylbenzene, and xylenes
CARB = California Air Resources Board
EDB = ethylene dibromide
G = glass
HCl = hydrochloric acid
L = liter
mL = milliliter
pH = potential hydrogen
VOC = volatile organic compound

1. All containers must have Teflon-lined seals.
2. (Teflon-lined septa for volatile organic analysis [VOA] vials).
3. Sample preservation will be completed in the field immediately upon sample collection.
4. When only one holding time is given, it implies total holding time from sampling until analysis.

Table 3-2. Analytical Method, Preservation, and Holding Time Requirements - Drinking Water Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	Analytical Group	Analytical and Preparation Method/SOP Reference	Sample Volume	Container (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Water	BTEX – EPA 524.2	Preparation: EPA 524.2 Analysis: EPA 524.2	40 mL	3 X 40 mL VOA with Teflon® septa	HCL to pH <2 Cool at 0-4°C	14 days for analysis
Water	EDB – EPA 504.1	Preparation: EPA 504.1 Analysis: EPA 504.1	40 mL	3 X 40 mL VOA with Teflon® septa	Cool at 0-4°C	14 days for analysis

Acronyms and Abbreviations:
< = less than
°C = degrees Celsius
BTEX = benzene, toluene, ethylbenzenes, and xylenes
EDB = ethylene dibromide
EPA = United States Environmental Protection Agency
HCl = hydrochloric acid
mL = milliliter
NA = not applicable
pH = potential hydrogen
SOP = standard operating procedure
SVOC = semi volatile organic compound
VOA = volatile organic analysis

Table 3-3. Analytical Method, Preservation, and Holding Time Requirements – Soil Vapor Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	Analytical Group	Analytical and Preparation Method/SOP Reference	Sample Volume	Container (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Vapor	VOCs EPA TO15	Preparation: EPA TO15 Analysis: EPA TO15	1 L	1 L Bottle Vac Canister; 1L for Vapor	NA	30 days for analysis
Vapor	APH – Method MA DEP	Preparation: Method MA DEP Analysis: Method MA DEP	1 L	1 L Bottle Vac Canister	NA	28 days for analysis
Vapor	Fixed Gases – ASTM D2504	Preparation: ASTM D2504 Analysis: ASTM D2504	1 L	1 L Bottle Vac Canister	NA	30 days for analysis
Vapor	CARB 422	Preparation: CARB 422 Analysis: CARB 422	1 L	1 L Bottle Vac Canister	NA	30 days for analysis

***Acronyms & Abbreviations**:*
APH = air-phase petroleum hydrocarbon
ASTM = ASTM International
CARB = California Air Resources Board
EPA = U.S. Environmental Protection Agency
L = liter
MA DEP = Massachusetts Department of Environmental Protection
NA = not applicable
SOP = standard operating procedure
TPH = total petroleum hydrocarbon
VOC = volatile organic compound

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**VOCs, BTEX, and APH**					
Analytical Method	**Methods 524.2, MA DEP, and TO15**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicators**	**Measurement Performance Criteria**
Internal standards	Every field sample and QC samples	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Correct problem, then re- reanalyze affected samples.	Lab Manager/Analyst	Bias	RT within ±30 seconds and area count within -50% to +100%
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/Analyst	Representativeness	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 2 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**VOCs, BTEX, and APH**					
Analytical Method	**EPA Methods 524.2, MA DEP, and TO15**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicators**	**Measurement Performance Criteria**
MS/MSD for all analytes	One MS/MSD pair per preparation batch per matrix *Not performed on vapor samples	EPA 524.2 and 504.1, MA DEP: LCS control limits specified by laboratory SOP	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/Analyst	Precisions and Bias	EPA 524.2 and 504.1, MA DEP: LCS control limits specified by laboratory SOP
LCS or LCS/LCSD pair for all analytes	One LCS or LCS/LCSD pair per preparation batch per matrix	EPA 524.2 and 504.1, MA DEP: LCS control limits specified by laboratory SOP TO15: LCS control limits specified in the DOD QSM	Correct problem, then re-reanalyze the LCS and all associated batch samples	Lab Manager/Analyst	Precisions and Bias	EPA 524.2 and 504.1, MA DEP: LCS control limits specified by laboratory SOP TO15: LCS control limits specified in the DOD QSM

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 3 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**VOCs, BTEX, and APH**					
Analytical Method	**EPA Methods 524.2, MA DEP, and TO15**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicators**	**Measurement Performance Criteria**
Surrogate standards	Every field sample and QC sample	EPA 524.2 and 504.1: Surrogate recovery acceptance criteria specified in laboratory SOP TO15: Specified in DOD QSM	Correct problem, then re-reanalyze all affected samples	Lab Manager/Analyst	Bias	EPA 524.2 and 504.1: Surrogate recovery acceptance criteria specified in laboratory SOP TO15: Specified in DOD QSM Version 5.0
Sample duplicate	Every 20 samples	TO15: Specified in DOD QSM MA DEP: Surrogate recovery acceptance criteria specified in laboratory SOP	NA	Lab Manager/Analyst	Bias	TO15: Specified in DOD QSM MA DEP: Surrogate recovery acceptance criteria specified in laboratory SOP
MDL study	Initial setup *Not run for MA APH	Detection limits established will be below the LOQs	Correct problem, then repeat the MDL study	Lab Manager/Analyst	Sensitivity	

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 4 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**VOCs, BTEX, and APH**					
Analytical Method	**EPA Methods 524.2, MA DEP, and TO15**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
LOD study	Initial setup and quarterly LOD verification *Not run for MA APH	Signal to noise ratio at the LOD will be greater than 3 and meet method requirements.	Correct problem, then repeat detection limit study and LOD verification at a higher concentration, or pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration in accordance with DoD QSM requirements.	Lab Manager/Analyst	Sensitivity	
LOQ study	Annually and quarterly LOQ verification	LOQ will be greater than LOD and within calibration range. Laboratory procedure for establishing the LOQ will empirically demonstrate precision and bias at the LOQ LOQ>LOD>DL		Lab Manager/Analyst	Sensitivity	

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 5 of 5)

Acronyms and Abbreviations:
% = percent
APH = air-phase petroleum hydrocarbon
DL = detection limit
DoD = U.S. Department of Defense
EPA = U.S. Environmental Protection Agency
LCS = laboratory control sample
LCSD =laboratory control sample duplicate
LOD = limit of detection
LOQ = limit of quantitation
MA APH = Massachusetts Air-Phase Petroleum Hydrocarbon
MA DEP = Massachusetts Department of Environmental Protection
MDL = method detection limit
MS = matrix spike
MSD = matrix spike duplicate
NA = not applicable
QC = quality control
QSM = Quality Systems Manual
RL = reporting limit
RPD = relative percent difference
RT = retention time
SVOC = semi volatile organic compound
VOC = volatile organic compound

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**EDB, TPH, Fixed Gases**					
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and >1/10 amount detected in project samples or 1/10 the regulatory limit (whichever is greater)	Correct problem, then re-extract and reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/Analyst	Representativeness	No target analytes detected greater than one-half RL and >1/10 amount detected in project samples or 1/10 the regulatory limit (whichever is greater)
MS/MSD for all analytes	One MS/MSD pair per preparation batch per matrix *Not performed on vapor samples	EPA 504.1, MA DEP: Laboratory in-house LCS control limits RPD less than 30% between MS and MSD	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples	Lab Manager/Analyst	Precisions and Bias	EPA 504.1, MA DEP: Laboratory in-house LCS control limits RPD less than 30% between MS and MSD

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 2 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**EDB, TPH, Fixed Gases**					
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
LCS or LCS/LCSD pair for all analytes	One LCS or LCS/LCSD pair per preparation batch per matrix	EPA 524.2 and 504.1, MA-DEP: Laboratory in-house LCS control limits	Correct problem, then re-extract and reanalyze the LCS and all associated batch samples	Lab Manager/Analyst	Precisions and Bias	EPA 524.2 and 504.1, MA-DEP: Laboratory in-house LCS control
Surrogate standards	Every field sample and QC sample *Not added to CARB422 or fixed gasses	EPA 524.2 and 504.1, MA-DEP: Laboratory in-house surrogate acceptance criteria	Correct problem, then re-extract and reanalyze all affected samples	Lab Manager/Analyst	Bias	EPA 524.2 and 504.1, MA-DEP: Laboratory in-house surrogate acceptance criteria

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 3 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**EDB, TPH, Fixed Gases**					
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
Confirmation of positive results using second column or second detector	All positive results must be confirmed	Same calibration and QC requirements as for initial or primary column analysis. RPD between primary and second column results less than 40%	NA	Lab Manager/Analyst	Precision	RPD between primary and second column results less than 40%
MDL study	Initial setup *Not run for fixed gasses	Detection limits established will be below the LOQs	Correct problem, then repeat the MDL study in accordance with DoD QSM requirements	Lab Manager/Analyst	Sensitivity	

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 4 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**EDB, TPH, Fixed Gases**					
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
LOD study	Initial setup and quarterly LOD verification *Not run for CARB422 or fixed gasses	Signal to noise ratio at the LOD will be greater than 3 and meet method requirements.	Correct problem, then repeat detection limit study and LOD verification at a higher concentration, or pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration per DoD QSM	Lab Manager/Analyst	Sensitivity	
LOQ study	Annually and quarterly LOQ verification	LOQ will be greater than LOD and within calibration range. Laboratory procedure for establishing the LOQ will empirically demonstrate precision and bias at the LOQ LOQ>LOD>DL		Lab Manager/Analyst	Sensitivity	

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 5 of 5)

Acronyms and Abbreviations:
% = percent
ASTM = ASTM International
CARB=California Air Resources Board
DoD = U.S. Department of Defense
EDB = ethylene dibromide
EPA = U.S. Environmental Protection Agency
EPH = extractable petroleum hydrocarbon
LCS = laboratory control sample
LCSD =laboratory control sample duplicate
LOD = limit of detection
LOQ = limit of quantitation
MA DEP = Massachusetts Department of Environmental Protection
MDL = method detection limit
MS = matrix spike
MSD = matrix spike duplicate
NA = not applicable
QC = quality control
QSM = Quality Systems Manual
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure
TPH = total petroleum hydrocarbon
VPH = volatile petroleum hydrocarbon

Table 3-6. Field Instrument Quality Control
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Field Equipment	Calibration Verification Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Photoionization Detector	Check calibration to 100 parts per million isobutylene	Once per day before first use	± 5% of standard value	Recalibrate	Sundance Field Personnel	Manufacturer's Operation Manual
Horiba	Check calibration for petroleum hydrocarbons and CO_2 against a calibration standard of known concentrations in a premixed gas cylinder	Once per day before first use	± 10% of standard value	Recalibrate	Sundance Field Personnel	Manufacturer's Operation Manual – modified per WP
	Check calibration for O_2 against atmospheric concentrations	Once per day before first use	O_2>22%	Recalibrate	Sundance Field Personnel	Manufacturer's Operation Manual – modified per WP
YSI 556 Multi-Probe System Water Quality Meter	Check calibration for multi-probe meters against manufacturer provided calibration standards.	Once per day before first use	Manufacturer's Standard	Recalibrate	Sundance Field Personnel	Manufacturer's Operation Manual – modified per WP

Acronyms and Abbreviation
> = greater than
% = percent
CO_2 = carbon dioxide
O_2 = oxygen
SOP = standard operating procedure
Sundance = Sundance Consulting, Inc.
WP = Work Plan
YSI = Yellow Springs Instruments

Table 3-7. Laboratory Instrument Quality Control – Drinking Water Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed.	Water samples	Ion source, injector liner, column, column flow, purge lines, purge flow, trap	Prior to initial calibration and/or as necessary	Acceptable tune and calibration or CCV	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	TestAmerica Analyst and Laboratory Manager	TestAmerica SA-VO-002, Rev. 5
MS	Change the injection port liner, column ferrule, and autosampler syringe as needed. Liners should be changed when recent sample analyses predict a problem with chromatographic performance. The autosampler should be cleaned periodically. This includes turret cleaning and cleaning or replacing the syringe.	Water Samples	injection port liner, column ferrule, and autosampler syringe	Prior to initial calibration and/or as necessary	Acceptable tune and calibration or CCV	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	TestAmerica Analyst and Laboratory Manager	TestAmerica SA-SG-060, Rev. 12

Table 3-7. Laboratory Instrument Quality Control – Drinking Water Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 2 of 2)

Acronyms and Abbreviations:
BFB = bromofluorobenzene
CCV = continuing calibration verification
GC/MS = gas chromatography/mass spectrometry
SOP = standard operating procedure
VOC = volatile organic compound

Table 3-8. Laboratory Instrument Quality Control – Soil Vapor Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Daily/regular as specified	Air samples	Instrument operating parameters	Daily	Per SOP	Recalibrate/ stop for service on failure	ALS Laboratory Analyst and Laboratory Manager	VOA-TO-15 Rev 22 VOA-MAPH Rev 9
GC	Daily during use.	Air/gas samples	Instrument operating parameters	Daily	Per SOP	Recalibrate/ stop for service on failure	ALS Laboratory Analyst and Laboratory Manager	VOA-EPA 3C Rev 13 SVO-CARB422 Rev 5

Acronyms and Abbreviations:
ALS = ALS Environmental Laboratory, Inc.
CARB = California Air Resources Board
EPA = U.S. Environmental Protection Agency
GC = gas chromatography
GC = gas chromatography
MS = mass spectrometry
SOP = standard operating procedure
VOA = volatile organic compound

Table 3-9. Analytical Instrument Calibration - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	**Drinking Water and Soil Vapor**				
Analytical Group	**VOCs, BTEX, and APH**				
Analytical Method	**EPA Methods 524.2, MA DEP, and TO15**				
Instrument	**Calibration Procedure**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**
GC/MS	Check of mass spectral ion intensities (tuning procedure) using bromofluorobenzene	Prior to initial calibration and meet frequency requirements specified in the method	Must meet the method requirements before samples are analyzed	Retune instrument and verify the tune acceptability, rerun the affected samples	Lab Manager/Analyst
	Five-point initial calibration for target analytes, lowest calibration standard at or near the LOQ in accordance with DoD QSM requirements	Initial calibration prior to sample analysis	TO15 and MA DEP: RSD is less than 30%per method requirements 524.2: RSD is less than 20% per method requirements	Correct problem, then rerun initial calibration in accordance with DoD QSM/method requirements	Lab Manager/Analyst

Table 3-9. Analytical Instrument Calibration - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 2 of 3)

Matrix	**Drinking Water and Soil Vapor**				
Analytical Group	**VOCs, BTEX, and APH**				
Analytical Method	**EPA Methods 524.2, MA DEP, and TO15**				
Instrument	**Calibration Procedure**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**
GC/MS	Second-source calibration verification in accordance with DoD QSM requirements	Once per five-point initial calibration	EPA 524.2: Less than 30% difference for all target analytes in accordance with method requirements. MA DEP: Less than 25% difference for all target analytes in accordance with method requirements	Correct problem, then rerun second source calibration verification in accordance with DoD QSM/method requirements	Lab Manager/Analyst
	Daily calibration verification in accordance with DoD QSM requirements	Before sample analysis and every 12 hours of analysis	EPA 524.2 and TO15: Less than 30% difference for all target analytes in accordance with method requirements MA DEP: Less than 25% difference for all target analytes per method requirements.	Correct problem, then rerun calibration verification in accordance with DoD QSM/method requirements	Lab Manager/Analyst
	Breakdown check	Before sample analysis and every 12 hours of analysis		Correct problem, then rerun breakdown check	Lab Manager/Analyst

Table 3-9. Analytical Instrument Calibration - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 3 of 3)

Acronyms and Abbreviations:
% = percent
APH = air phase petroleum hydrocarbon
DDT = dichlorodipheyl trichloroethane
DoD = U.S. Department of Defense
EPA = U.S. Environmental Protection Agency
GC/MS = gas chromatography/mass spectrometry
LCS = laboratory control sample
LOQ = limit of quantitation
MA APH = Massachusetts Air-Phase Petroleum Hydrocarbon
MA DEP = Massachusetts Department of Environmental Protection
QSM = Quality Systems Manual
RSD = relative standard deviation
SVOC = semi volatile organic compound
VOC = volatile organic compound

Table 3-10. Analytical Instrument Calibration- Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	**Drinking Water and Soil Vapor**				
Analytical Group	**EDB, TPH, Fixed Gases**				
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**				
Instrument	**Calibration Procedure**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**
GC	Minimum five-point initial calibration for target analytes, lowest calibration standard at or near the LOQ in accordance with DoD QSM requirements Stable Isotope: perform external calibration of working standard per laboratory SOPs	Initial calibration prior to sample analysis	EPA 504.1: RSD less than or equal to 20% for all target analytes in accordance with DoD QSM requirements MA DEP: RSD less than 25% for all target analytes per method requirements	Correct problem, then rerun initial calibration in accordance with DoD QSM requirements.	Lab Manager/Analyst
	Second-source calibration verification	Once per five-point initial calibration	EPA 504.1: Less than 20% of expected values from the initial calibration for all target analytes in accordance with DoD QSM requirements MA DEP: Less than 25% of expected values from the initial calibration for all target analytes per method requirements	Correct problem, then rerun second source calibration verification in accordance with DoD QSM requirements.	Lab Manager/Analyst

Table 3-10. Analytical Instrument Calibration - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 2 of 2)

Matrix	**Drinking Water and Soil Vapor**				
Analytical Group	**EDB, TPH, Fixed Gases**				
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**				
Instrument	**Calibration Procedure**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**
GC	Daily calibration verification.	EPA 524.2 and 504.1, MA DEP, Before sample analysis and at frequency specified in the method	MA DEP: Less than 25% of expected values from the initial calibration for all target analytes per method requirements	Correct problem, then rerun calibration verification in accordance with DoD QSM requirements	Lab Manager/ Analyst

Acronyms and Abbreviations:
% = percent
ASTM = ASTM International
DoD = U.S. Department of Defense
EDB = ethylene dibromide
EPA = U.S. Environmental Protection Agency
EPH = extractable petroleum hydrocarbon
GC = gas chromatography
LOQ = limit of quantitation
MA DEP = Massachusetts Department of Environmental Protection
QSM = Quality Systems Manual
RSD = relative standard deviation
SOP = standard operating procedure
TPH = total petroleum hydrocarbon
VPH = volatile petroleum hydrocarbon

4 DATA VALIDATION AND USABILITY

4.1 Analytical Data Review, Verification, and Validation

The laboratory analyst who generates the analytical data will have primary responsibility for the correctness and completeness of data. Each step of this verification and review process will involve the evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of data is essential in ensuring that data of known quality is consistently generated. All data generated and reduced will follow well-documented in-house protocols.

4.1.1 Level 1: Technical or Peer Data Review

Analysts will review the quality of their work based on an established set of guidelines, including the QC criteria established in each method, in this QAPjP, and as stated within the laboratory QA manual (Attachment C). This review will, at a minimum, ensure that the following conditions have been met:

- Sample preparation information is correct and complete.
- Analysis information is correct and complete.
- Appropriate SOPs have been followed.
- Calculations are verified.
- There are no data transposition errors.
- Analytical values are correct and complete.
- QC samples results are within established control limits.
- Blank results are within appropriate QC limits.
- LCS results are within appropriate QC limits.
- Special sample preparation and analytical requirements have been met.
- Documentation is complete; for example, any anomalies and holding times have been documented and forms have been completed.

4.1.2 Level 2: Technical Data Review

A supervisor or data review specialist whose function is to provide an independent review of data packages will perform this review. This review will also be conducted according to an established set of guidelines and will be structured to verify the Level 1 data review. This review will, at a minimum, ensure that the following conditions have been met:

- Appropriate laboratory SOPs are followed.
- Calibration data are scientifically sound and appropriate to the method.
- QC samples results are within established guidelines.

- Qualitative identification of contaminants is correct.
- Manual integrations are justified and documented.
- Quantitative results and calculations are correct.
- Data is qualified correctly.
- Documentation is complete.
- The data package is complete and complies with contract requirements.

The Level 2 review will be structured so that all calibration data and QC sample results are reviewed and all of the analytical results from at least 10% of the samples are checked back to the sample preparation and analytical bench sheets. If no problems are found with the data package, the review will be considered complete. If discrepancies are identified, additional data evaluation is required.

4.1.3 Level 3: Administrative Quality Assurance Data Review

The laboratory QA Manager will review 10% of all data packages. This review should be similar to the review as provided in Level 2, except that it will provide a total overview of the data package to ensure its consistency and compliance with project requirements. All errors noted will be corrected and documented.

4.2 Analytical Data Verification and Validation

Sundance will subcontract a third party data validator utilizing established data validation procedures (manually or automated) to perform EPA 100% review and Level III data validation. The review will be performed for drinking water, and soil vapor analytical data obtained from each of the field tasks.
The data review will be performed using the QC criteria specified in the following analytical method and data validation guidelines:

- Project-specific QAPjP
- *DoD Quality Systems Manual for Environmental Laboratories, Version 5.0* (July 2013)
- *USEPA Test Methods for Evaluating Solids Waste, Physical/Chemical Methods* (SW 846, 2006 and updates)
- *USEPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-15, Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* (January 1999)
- MA DEP, *Method for the Determination of Extractable Petroleum Hydrocarbons (EPH)* (May 2004a)
- MA DEP, *Method for the Determination of Volatile Petroleum Hydrocarbons (VPH)* (May 2004b)
- MA DEP, *Method for the Determination of Air-Phase Petroleum Hydrocarbons (APH)* (December 2009)

- American Public Health, Association, American Water Works Association, and Water Environment Federation, *Standard Methods for the Examination of Water and Wastewater, 21st Edition* (2005)
- *USEPA Contract Laboratory Program, National Functional Guidelines for Superfund Organic Methods Data Review* (August 2014)
- *USEPA Contract Laboratory Program, National Functional Guidelines for Inorganic Superfund Data Review, Final* (August 2014)

The following QC elements will be included in the EPA 100% review and Level III data validation:

- Sample extraction and analysis holding times
- Laboratory method blanks
- Surrogate spike recoveries
- LCS/LCSD recoveries
- MS/MSD recoveries
- Laboratory Duplicate, LCS/LCSD and MS/MSD Relative percent differences (RPD)
- Initial calibrations
- Continuing and initial calibration verifications
- Trip, rinse, and ambient field blank results
- Field duplicate sample precision
- For GCMS:
    - Instrument Tune
    - Internal Standards
- Serial Dilutions

Data will be validated and flagged with the following data qualifiers as applicable:

- **J+ qualifier** denotes the analyte was positively identified, but the associated numerical value is estimated with a potential high bias.
- **J- qualifier** denotes the analyte was positively identified, but the associated numerical value is estimated with a potential low bias.
- **U qualifier** denotes the analyte was analyzed for, but was not detected above the MDL.

- **UJ qualifier** denotes that the analyte was not detected above the reported sample LOQ; however, the reported LOQ is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

- **R qualifier** denotes the data are unusable due to deficiencies in the ability to analyze the sample and meet QC criteria and DQOs.

As a result of the Level III data validation process, EPA qualifiers will be generated and applied to the affected sample results that exceeded the established QC criteria. EPA 100% review and level III data validation findings will be summarized and documented with each monitoring report.

4.3 Reconciliation with User Requirements

Based on data review and data qualification, the Data Validator will determine if the project DQOs have been met, and data completeness will be calculated. To reconcile the collected data with project DQOs and to establish and document data usability, the data will be reviewed against data quality indicators discussed below.

The Data Validator will prepare a data quality assessment (DQA) report for each of the monitoring events. The DQA report will document:

- Implementation of sampling design and analysis according to the approved QAPjP (or sample completeness and representativeness)

- Proper frequency of field QC samples and the adequacy of field decontamination procedures

- Accuracy and precision of the data

- Data comparability, if applicable

- Data usability for project decisions

4.3.1 Data Quality Indicators

This section defines the data quality indicators and their use for assessment of data quality. These indicators include the PARCC parameters of precision, accuracy, representiveness, comparability and completeness.

4.3.1.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions. The following equation illustrates the method for calculating relative percent difference (RPD) to assess a method's precision:

Precision as RPD	=	$\frac{\text{Absolute (Result – Duplicate Result)}}{\text{Average (Result + Duplicate Result)}}$	x	100%

The laboratory uses MS/MSD samples to assess the precision of analytical procedures. According to USACE requirements, analytical laboratories perform MS/MSD on the project samples to determine whether matrix interferences may be present.

In addition, LCS/LCSD samples can be used to determine analytical method precision when MS/MSD samples are not practical due to the nature of sample or analytical method used. Laboratories will use precision limits specified in the DoD QSM for both LCS and MS analyses (DoD, 2013). When precision limits are not available in the DoD QSM, laboratories may use statistically-based acceptability limits for RPDs established for each method of analysis and sample matrix. The laboratory will review the QC samples to ensure that internal QC data achieve limits of acceptability. Any suspect trends will be investigated and corrective actions taken.

4.3.1.2 Accuracy

Accuracy measures the bias of an analytical system by comparing the difference of a measurement with a reference value. The percent recovery of an analyte, which has been added to the environmental samples at a known concentration before extraction and analysis, provides a quantitation tool for analytical accuracy. The spiking solutions used for accuracy determinations are not used for instrument calibrations. The following equation illustrates how accuracy is evaluated:

Accuracy as Percent Recovery	=	$\frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Spiked Sample True Value}}$	x	100%

Percent recoveries for MS, MSD, and LCS serve as a measure of analytical accuracy. Surrogate standards are added to all samples, blanks, MS, MSD, and LCS analyzed for gas chromatography and mass spectrometry analytical methods to evaluate accuracy of the method and help to determine matrix interferences.

Laboratories will use LCS limits specified in the DoD QSM for both LCS and MS analyses (DoD 2013). When LCS limits are not available in the DoD QSM, the laboratory may use in-house, statistically-based, control limits or control limits specified in EPA methods.

4.3.1.3 Representativeness

Unlike precision and accuracy, which can be expressed in quantitative terms, representativeness is a qualitative parameter. Representativeness is the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. A qualitative parameter depends on proper design of the sampling program.

Field personnel will be responsible for ensuring that samples are representative of field conditions by collecting and handling samples according to the approved QAPjP and WP. Errors in sample collection, packaging, preservation, or COC procedures may result in samples being judged non-representative and may form a basis for rejecting the data.

Data generated by the laboratory must be representative of the laboratory database of accuracy and precision measurements for analytes in different matrices. Laboratory procedures for sample preparation will ensure that aliquots used for analysis are representative of the whole sample. Aliquots to be analyzed for volatile parameters (if any) will be removed before the laboratory composites/homogenizes the samples, to avoid losing volatile compounds during mixing.

4.3.1.4 Comparability

Comparability is a qualitative parameter expressing the confidence where one data set can be compared with another, whether it was generated by a single laboratory or during laboratory studies. The use of standardized field and analytical procedures ensures comparability of analytical data.

Sample collection and handling procedures will adhere to EPA-approved protocols. Laboratory procedures will follow standard analytical protocols, use standard units and standardized report formats, follow the calculations as referenced in approved analytical methods, and use a standard statistical approach for QC measurements.

4.3.1.5 Completeness

Completeness goals for each sampling round are defined in the following section.

4.3.1.5.1 Contractual Completeness

The contractual completeness goal is set at 95% for all methods and is calculated as defined below. The following QC elements are evaluated for the purpose of determining completeness calculation:

- Holding time
- Laboratory blank contamination
- Initial calibration verification
- Continuing calibration verification
- LCSs

% Contract Completeness	=	$\frac{\text{Number of Unqualified Results*}}{\text{Number of Results Reported}}$	x	100%

** Determined by subtracting the results qualified based on contractual deficiencies from the total number of results*

4.3.1.5.2 Analytical Completeness

The analytical completeness goal is set at 90% for all methods and is calculated as defined below. The following QC elements will be considered analytical deficiencies for the purposes of the analytical completeness calculation:

- Holding time
- Laboratory blank contamination
- Field blank contamination (trip, equipment, ambient, and rinse)
- Initial calibration verification
- Continuing calibration verification

- LCS recovery
- MS recovery
- MS precision
- Surrogate recovery

% Analytical Completeness	=	Number of Unqualified Results* / Number of Results Reported	x	100%

** Determined by subtracting results qualified for any of the deficiencies from the total number of results.*

4.3.1.5.3 Technical Completeness

The technical completeness goal is set at 95% for all methods and is calculated as defined below. Results considered unusable (or rejected) for the intended purpose based on contractual or technical deficiencies will be included for the purposes of the technical completeness calculation:

% Technical Completeness	=	Number of Useable Results* / Number of Results Reported	X	100%

** Technical completeness (i.e., usability) will be determined by subtracting results rejected for any reason from the total number of results reported.*

4.3.2 Project-Required Reporting Limits – Sensitivity

Following the DoD QSM requirements, the laboratory will determine the method detection limits (MDL) for each method, instrument, analyte, and matrix by using the procedure described in Title 40 Code of Federal Regulations Part 136B. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

Following MDL studies, the laboratory will establish the reporting limit or LOQ for each method, analyte, matrix, and instrument in accordance with the DoD QSM requirements. The LOQ is the lowest concentration of a substance that produces a quantitative result within specific limits of precision and bias. The laboratory will perform LOQ verifications to verify precision and bias at the LOQ. The LOQ is greater than the LOD and must be within the calibration range prior to sample analysis. For this project, the laboratory will report positive results down to the MDL and results between the DL and LOQ will be flagged with a J-qualifier and reported as estimated data.

5 ASSESSMENT AND OVERSIGHT

Performing assessments and conducting QA oversight of project activities are vital to verifying that project objectives are being met and assuring the continued quality of the work performed. Assessments will take the form of field surveillances. QA oversight will regularly be performed onsite and is intended to be an interactive part of the field work performed. QA oversight will be performed by the Sundance Project Technical Lead, or designee. QA oversight includes inspections of work performed, verification of field documentation, and site walk-downs.

5.1 Quality Assurance Assessments

Independent assessments shall be planned and conducted to measure item and service quality, evaluate the adequacy of work performance, and promote improvement. The purpose of these assessments is to evaluate the performance of work processes with regard to regulatory, contract, and project requirements and expectations of the client. The group performing independent assessments shall have sufficient authority and freedom from the Sundance project staffing and management line to carry out its responsibilities. Persons conducting independent assessments must be technically qualified and knowledgeable in the areas assessed.

The independent assessment program may include periodic field surveillances of field activities (e.g., soil vapor and drinking water sampling, etcetera). Special emphasis will focus on areas with the highest risk and the greatest benefit from improvement. The surveillance processes will consist of monitoring or observing an item, activity, system, or process to verify that it conforms to specified requirements. These types of assessments are intended to facilitate the frequent monitoring of work in progress to determine and document compliance with established requirements and procedures.

5.2 Quality Assurance Oversight

QA oversight will be performed onsite and is intended to be an interactive part of the field work performed. QA oversight will be performed by the Sundance QA Lead or designee. QA oversight includes inspections of work performed, verification of field documentation, and site walk-downs.

5.2.1 Inspections

Inspection activities will be used to monitor project activities and materials to ensure compliance with established requirements. The objective of inspections is to determine whether the properties, composition, and performance of activities or materials are within established requirements. Inspections shall be performed periodically during the work process to prevent unintended use or installation, to provide monitoring, to minimize delays in work, and to identify nonconformances while they are still correctible without impacting work.

5.2.2 Verification of Field Documentation

Field documentation (e.g., Field Activity Daily Logs, Sample Collection Logs, etcetera) will be reviewed and verified for accuracy and completeness on a regular basis. This verification process is an informal process performed as part of report preparation; allowing for the quick and efficient correction of documentation deficiencies.

5.2.3 Site Walk-downs

Site walk-downs are informal observations of field work being performed. The intent of a site walk-down is to verify that the work is being performed as planned in a safe and orderly manner. Any deficiencies

identified during a walk-down are immediately pointed out to the field crew and corrected. Walk-downs are performed on a daily basis by the Technical Lead/SSHO, but may also be performed by the Sundance PM, or any other senior Sundance personnel.

5.3 Nonconformances and Response Actions

Processes for detecting, preventing, and correcting quality problems are discussed in this section. Items and processes that do not meet established criteria shall be identified, controlled, and corrected, as applicable. Personnel at all levels are responsible for identifying problems and process improvement opportunities and are encouraged to offer solutions.

5.3.1 Problem Identification and Reporting

It is the responsibility of all Sundance and subcontractor personnel to assess activities and inspect items used within the project to verify that each meets specified requirements and to document incidences of nonconforming items, activities, or conditions on a Nonconformance Report (NCR) (Attachment D). It is the responsibility of the project management staff to promptly report, respond to, and resolve nonconforming conditions and to foster a "no-fault" attitude that encourages the identification of nonconforming items and processes.

Personnel who identify a nonconforming condition that is potentially hazardous to workers, the public, or the environment or that jeopardizes the integrity of the program or project have the responsibility and authority to suspend work and report the condition to the responsible manager.

5.3.2 Control and Disposition of Nonconforming Items

Items that do not meet specified requirements, known as nonconforming items, shall be identified by marking, tagging, or other methods that do not adversely affect their end use. Nonconforming items shall be segregated, when practical, by placing them in a clearly identified and designated hold area until properly dispositioned. If segregation is impractical or impossible due to physical conditions, then other administrative controls and precautions should be employed to preclude inadvertent use of nonconforming items.

5.3.3 Nonconforming Activities

Activities or documentation identified as out of compliance with requirements shall be documented as a nonconformance for the purpose of identification of corrective actions and evaluation of the effect on the project objectives. When the integrity of the work is left in question, the work should be performed again, if possible. When not possible, limitations of the results of the work must be documented in the final report of the work.

5.3.4 Cause Analysis

Cause analysis will be performed whenever the understanding of the basic underlying cause is important to the prevention of similar or related problems or when the nonconformance relates to safety. The extent of the cause analysis should be based on the possible negative consequences of a repeat occurrence of a problem. A cause analysis will be used to gain an understanding of the deficiency, its causes, and the necessary corrective actions to prevent recurrence. This analysis should be a systematic process of investigation that uncovers the most basic cause. A summary of the cause analysis shall be documented on the NCR.

5.3.5 Corrective Actions

Responsible managers shall develop and document corrective actions, as applicable, for identified nonconformances. Corrective actions should be targeted at the primary causes of the problem rather than the resulting conditions or secondary causes. These actions shall be reviewed for adequacy and effectiveness in correcting the problem and approved by the PM or a designee.

5.3.6 Improvements and Efficiencies

It is important to identify and report process improvements and efficiency gains. Successful techniques and processes will be evaluated by the Sundance PM, or designee, to determine the potential for performance improvements in other areas or projects.

5.4 Reports to Management

Reports to management may include assessment reports, inspection reports, and NCRs.

5.4.1 Assessment Reports

Surveillance activities will be documented in surveillance reports. Surveillance reports will identify the project activities that were observed/reviewed, the associated requirements documents, and the results of the surveillances, including deficiencies identified and noteworthy practices. Surveillance reports will be prepared/approved by the Sundance Corporate Quality Control Manager (QCM) and presented to the PM within 30 days of performance. Surveillance checklists used during the performance of the surveillance may be included with the final surveillance report. A copy of the final surveillance report shall be placed in the project files.

5.4.2 Nonconformance Reports

Nonconformance reporting will include a description of the nonconforming item or activity, a summary of the corrective action to be taken, assignment of who is responsible for completing the corrective action, and verification that the corrective action is completed. Nonconformance reports will be tracked by the Sundance QA Manager and evaluated by the Sundance PM. A copy of the NCR shall be placed in the project files.

6 DATA MANAGEMENT

Data management is discussed in Section 6.0 of the WP to which this QAPjP is included as an appendix. The WP provides the data management process and procedures to be implemented for the field and for handling laboratory data generated from work activities in support of SVM and drinking water monitoring.

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ATTACHMENT A

LABORATORY ANALYTICAL METHOD REPORTING LIMITS

Table A-1. Method Reporting Limits – Drinking Water

Table A-2. Method Reporting Limits – Soil Vapor

Table A-1. Method Reporting Limits – Drinking Water (TestAmerica, Savannah, GA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Analytical Method	Analyte	CAS Number	Units	NMWQCC [1]	EPA MCL [2]	EPA Tap water		Project Screening Level [4]	Achievable Laboratory Limits [5]		
						RSL [3]	c/nc		LOQ	LOD	MDL
BTEX by EPA 524.2	Benzene	71-43-2	µg/L	10.00	5.00	4.500	c	5.00	0.500	0.250	0.0820
	Ethylbenzene	100-41-4	µg/L	750.00	700.00	15.000	c	700.00	0.500	0.250	0.0990
	m, p-Xylenes	179601-23-1	µg/L	NS	10,000.00	190.000	nc	10,000.00	0.500	0.250	0.0860
	o-Xylene	95-47-6	µg/L	NS	10,000.00	190.000	nc	10,000.00	0.500	0.250	0.0860
	Toluene	108-88-3	µg/L	750.00	1000.00	1100.000	nc	750.00	0.500	0.250	0.0860
EDB by EPA 504.1	Ethylene dibromide	1832-54-8	µg/L	0.10	0.05	0.075	c	0.05	0.0180	0.00500	0.00220

NOTES:
[1] NMWQCC standards per the New Mexico Administrative Code Title 20.6.2.3101A, Standards for Ground Water of 10,000 mg/L Total Dissolved Solids Concentration or Less (NMAC 2004).
For metals, the NMWQCC standard applies to dissolved metals and total mercury.
[2] EPA National Primary Drinking Water Regulations, Maximum Contaminant Levels and Secondary Maximum Contaminant Levels, Title 40CFR Part 141, 143 (May 2009).
[3] EPA Region 6 Regional Screening Levels for Tap water (June 2015) for hazard index = 1.0 for noncarcinogens and a 10^{-5} cancer risk level for carcinogens.
[4] The project screening level was selected to satisfy the requirements of the KAFB Hazardous Waste Permit No. NM9570024423 as the lowest of 1) NMWQCC standard or 2) EPA MCL.
If no MCL or NMWQCC standard exists for any analyte, then the project screening level will be the EPA Tap water RSL.
[5] Achievable laboratory limits are for TestAmerica Laboratories, Savannah, Georgia.

Acronyms and Abbreviations:
µg/L = Microgram(s) per liter
mg/L = Milligram(s) per liter
c/nc = Carcinogenic/ noncarcinogenic

Table A-1. Method Reporting Limits – Drinking Water (TestAmerica, Savannah, GA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 2 of 2)

CA = California
CAS = Chemical Abstracts Service
DL = Detection limit
EDB = ethylene dibromide
EPA = U.S. Environmental Protection Agency
LOD = Limit of detection
LOQ = Limit of quantitation
MCL = Maximum Contaminant Level
MDL = Method Detection Limit
NMWQCC = New Mexico Water Quality Control Commission
NS = no standard
RSL = regional screening level
VOC = volatile organic compound

Table A-2. Method Reporting Limits - Soil Vapor (ALS Global - Environmental, Simi Valley, CA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Analytical Group/ Method	Analyte	CAS Number	Units	Project Screening Level	Achievable Laboratory Limits		
					LOQ	LOD	DL
VOCs/TPH EPA TO-15	1,1,1-Trichloroethane	71-55-6	ppbv	Note 1	0.23	0.19	0.078
	1,1,2,2-Tetrachloroethane	79-34-5	ppbv	Note 1	0.18	0.15	0.055
	1,1,2-Trichloroethane	79-00-5	ppbv	Note 1	0.23	0.20	0.073
	1,1-Dichloroethane	75-34-3	ppbv	Note 1	0.31	0.27	0.099
	1,1-Dichloroethene	75-35-4	ppbv	Note 1	0.32	0.28	0.110
	1,2,4-Trichlorobenzene	120-82-1	ppbv	Note 1	0.17	0.15	0.054
	1,2,4-Trimethylbenzene	95-63-6	ppbv	Note 1	0.25	0.22	0.076
	1,2-Dibromoethane	106-93-4	ppbv	Note 1	0.16	0.14	0.052
	1,2-Dichlorobenzene	95-50-1	ppbv	Note 1	0.21	0.18	0.062
	1,2-Dichloroethane	107-06-2	ppbv	Note 1	0.31	0.27	0.099
	1,2-Dichloropropane	78-87-5	ppbv	Note 1	0.27	0.24	0.087
	1,3,5-Trimethylbenzene	108-67-8	ppbv	Note 1	0.25	0.22	0.081
	1,3-Butadiene	106-99-0	ppbv	Note 1	0.57	0.47	0.250
	1,3-Dichlorobenzene	541-73-1	ppbv	Note 1	0.21	0.19	0.062
	1,4-Dichlorobenzene	106-46-7	ppbv	Note 1	0.21	0.17	0.058
	2-Butanone (MEK)	78-93-3	ppbv	Note 1	4.20	0.37	0.180
	2-Hexanone	591-78-6	ppbv	Note 1	0.31	0.27	0.098
	4-Methyl-2-pentanone	108-10-1	ppbv	Note 1	0.31	0.27	0.098
	Acetone	67-64-1	ppbv	Note 1	5.30	2.30	0.810
	Benzene	71-43-2	ppbv	Note 1	0.39	0.35	0.130
	Benzyl Chloride	100-44-7	ppbv	Note 1	0.24	0.22	0.053
	Bromodichloromethane	75-27-4	ppbv	Note 1	0.19	0.16	0.056

Table A-2. Method Reporting Limits- Soil Vapor (ALS Global - Environmental, Simi Valley, CA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 2 of 4)

Analytical Group/ Method	Analyte	CAS Number	Units	Project Screening Level	Achievable Laboratory Limits		
					LOQ	LOD	DL
VOCs/TPH EPA TO-15	Bromoform	75-25-2	ppbv	Note 1	0.12	0.11	0.036
	Bromomethane	74-83-9	ppbv	Note 1	0.32	0.26	0.120
	Carbon Disulfide	75-15-0	ppbv	Note 1	4.00	0.34	0.120
	Carbon Tetrachloride	56-23-5	ppbv	Note 1	0.20	0.18	0.060
	Chlorobenzene	108-90-7	ppbv	Note 1	0.27	0.24	0.087
	Chloroethane	75-00-3	ppbv	Note 1	0.47	0.38	0.160
	Chloroform	67-66-3	ppbv	Note 1	0.26	0.23	0.087
	Chloromethane	74-87-3	ppbv	Note 1	0.61	0.48	0.180
	cis-1,2-Dichloroethene	156-59-2	ppbv	Note 1	0.32	0.28	0.100
	cis-1,3-Dichloropropene	10061-01-5	ppbv	Note 1	0.28	0.23	0.077
	Cyclohexane	110-82-7	ppbv	Note 1	0.73	0.62	0.210
	Dibromochloromethane	124-48-1	ppbv	Note 1	0.15	0.13	0.047
	Dichlorodifluoromethane (CFC 12)	75-71-8	ppbv	Note 1	0.25	0.19	0.086
	Ethyl Acetate	141-78-6	ppbv	Note 1	0.69	0.60	0.240
	Ethylbenzene	100-41-4	ppbv	Note 1	0.29	0.25	0.092
	Hexachlorobutadiene	87-68-3	ppbv	Note 1	0.12	0.11	0.033
	m,p-Xylenes	179601-23-1	ppbv	Note 1	0.58	0.50	0.170
	Methyl tert-Butyl Ether	1634-04-4	ppbv	Note 1	0.35	0.31	0.120
	Methylene Chloride	75-09-2	ppbv	Note 1	0.36	0.32	0.120
	Naphthalene	91-20-3	ppbv	Note 1	0.24	0.21	0.086
	n-Heptane	142-82-5	ppbv	Note 1	0.31	0.27	0.100
	n-Hexane	110-54-3	ppbv	Note 1	0.35	0.31	0.110
	o-Xylene	95-47-6	ppbv	Note 1	0.29	0.24	0.086
	Propene	115-07-1	ppbv	Note 1	0.73	0.58	0.200
	Styrene	100-42-5	ppbv	Note 1	0.29	0.26	0.088
	Tetrachloroethene	127-18-4	ppbv	Note 1	0.18	0.15	0.052
	Tetrahydrofuran (THF)	109-99-9	ppbv	Note 1	0.42	0.37	0.170

Table A-2. Method Reporting Limits- Soil Vapor (ALS Global - Environmental, Simi Valley, CA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 3 of 4)

Analytical Group/ Method	Analyte	CAS Number	Units	Project Screening Level	Achievable Laboratory Limits		
					LOQ	LOD	DL
VOCs/TPH EPA TO-15	Toluene	108-88-3	ppbv	Note 1	0.33	0.29	0.110
	trans-1,2-Dichloroethene	156-60-5	ppbv	Note 1	0.32	0.26	0.120
	trans-1,3-Dichloropropene	10061-02-6	ppbv	Note 1	0.28	0.23	0.088
	Trichloroethene	79-01-6	ppbv	Note 1	0.23	0.20	0.065
	Trichlorofluoromethane	75-69-4	ppbv	Note 1	0.22	0.19	0.076
	Trichlorotrifluoroethane	76-13-1	ppbv	Note 1	0.16	0.14	0.055
	Vinyl Acetate	108-05-4	ppbv	Note 1	3.60	1.50	0.460
	Vinyl Chloride	75-01-4	ppbv	Note 1	0.49	0.39	0.170
APH Method MA DEP	C5-C8 Aliphatic Hydrocarbons	NA	µg/m^3	Note 1	50.00	NA	NA
	C9-C12 Aliphatic Hydrocarbons	NA	µg/m^3	Note 1	25.00	NA	NA
	C9-C10 Aromatic Hydrocarbons	NA	µg/m^3	Note 1	6.30	NA	NA
Fixed Gases ASTM D2504	Oxygen	7782-44-7	%	Note 1	0.10	NA	NA
	Nitrogen	7727-37-9	%	Note 1	0.10	NA	NA
	Carbon Monoxide	630-08-0	%	Note 1	0.10	NA	NA
	Carbon Dioxide	124-38-9	%	Note 1	0.10	NA	NA
	Methane	74-82-8	%	Note 1	0.10	NA	NA
EDB CARB 422	Ethylene Dibromide	1832-54-8	ppbv	Note 1	0.50	NA	0.180

Notes:
Project comparison limits not established.
In accordance with the U.S. Department of Defense Quality Systems Manual requirements, the most current version of the EPA methods will be implemented for each sampling event.

Table A-2. Method Reporting Limits- Soil Vapor (ALS Global - Environmental, Simi Valley, CA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 4 of 4)

Acronyms and Abbreviations:
% = percent
APH = air- phase petroleum hydrocarbon
ASTM = ASTM International
CA = California
CAS = Chemical Abstract Service
DL = Detection Limit
MDL = method detection limit
EPA = U.S. Environmental Protection Agency
LOD = limit of detection
LOQ = limit of quantitation
MA DEP = Massachusetts Department of Environmental Protection
NE = not established
ppbv = parts per billion by volume
RL = reporting limit
TPH = total petroleum hydrocarbon
VOC = volatile organic compound

ATTACHMENT B

LABORATORY METHOD CONTROL LIMITS

B-1. Laboratory Control Limits – Drinking Water

B-2. Laboratory Control Limits – Soil Vapor

Table B-1. Method Reporting Limits - Drinking Water

METHOD	ANALYTE	CAS No.	MATRIX	MDL	MRL	UNITS	Accuracy (LCS %Rec)	Matrix Spike (%Rec.)	Precision (% RPD)		DOD LOD	DOD LOQ	UNITS
504.1	Ethylene Dibromide (EDB)	106-93-4	DW	0.003	0.01	ug/L	70-130	65-135	NA		0.007	0.01	ug/L
524.2	Benzene	71-43-2	DW	0.022	0.5	ug/L	70-130	NA	NA		0.08	0.5	ug/L
524.2	Ethylbenzene	100-41-4	DW	0.023	0.5	ug/L	70-130	NA	NA		0.08	0.5	ug/L
524.2	m,p-Xylenes	179601-23-1	DW	0.045	0.5	ug/L	70-130	NA	NA		0.16	0.5	ug/L
524.2	o-Xylene	95-47-6	DW	0.023	0.5	ug/L	70-130	NA	NA		0.08	0.5	ug/L
524.2	Toluene	108-88-3	DW	0.05	0.5	ug/L	70-130	NA	NA		0.2	0.5	ug/L

Acronyms and Abbreviations:

% = percent
ug/L = microgram(s) per liter
DoD = U.S. Department of Defense
DW = drinking water
EDB = ethylene dibromide
LCS = laboratory control sample
LOD = limit of detection
LOQ = limit of quantitation
MDL = method detection limit
MRL = method reporting limit
NA = not applicable
RPD = relative percent difference

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client:

Client Sample ID: Lab Control Sample ALS Project ID: P1504757

Client Project ID: Kirtland AFB / 140705 ALS Sample ID: P151119-LCS

Test Code:	Massachusetts APH, Revision 1, December 2009	Date Collected:	NA
Instrument ID:	Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13	Date Received:	NA
Analyst:	Evelyn Alvarez	Date Analyzed:	11/19/15
Sample Type:	1.0 L Bottle-Vac™	Volume(s) Analyzed:	0.125 Liter(s)

Test Notes:

Compound	Spike Amount µg/m³	**Result µg/m³**	**% Recovery**	ALS Acceptance Limits	**Data Qualifier**
C5 - C8 Aliphatic Hydrocarbons	216	**200**	**93**	70-130	
C9 - C12 Aliphatic Hydrocarbons	202	**197**	**98**	70-130	
C9 - C10 Aromatic Hydrocarbons	422	**388**	**92**	70-130	

Verified By:_____Date:_____

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client:

Client Sample ID: **Method Blank**
Client Project ID: **Kirtland AFB / 140705**

ALS Project ID: P1504757
ALS Sample ID: P151119-MB

Test Code:	Massachusetts APH, Revision 1, December 2009	Date Collected:	NA
Instrument ID:	Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13	Date Received:	NA
Analyst:	Evelyn Alvarez	Date Analyzed:	11/19/15
Sample Type:	1.0 L Bottle-Vac™	Volume(s) Analyzed:	0.40 Liter(s)

Test Notes:

Compound	**Result µg/m³**	MRL µg/m³	**Data Qualifier**
C_5 - C_8 Aliphatic Hydrocarbons[1,2]	50	50	**U**
C_9 - C_{12} Aliphatic Hydrocarbons[1,3]	25	25	**U**
C_9 - C_{10} Aromatic Hydrocarbons	6.3	6.3	**U**

Significant non-petroleum related peaks (i.e. halogenated, oxygenated, terpenes, etc.) are subtracted from the hydrocarbon range areas when present.

[1]Hydrocarbon Range data from total ion chromatogram excluding any internal/tuning standards eluting in that range.

[2]C_5-C_8 Aliphatic Hydrocarbons exclude the concentration of Target APH analytes eluting in that range.

[3]C_9-C_{12} Aliphatic Hydrocarbons exclude concentration of Target APH Analytes eluting in that range and concentration of C_9-C_{10} Aromatic Hydrocarbons.

ND = Compound was analyzed for, but not detected above the laboratory reporting limit.

MRL = Method Reporting Limit - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client:

Client Project ID: Kirtland AFB / 140705

ALS Project ID: P1504757

1,2-Dibromoethane

Test Code: CARB 422 Modified
Instrument ID: HP5890 II/GC21/ECD
Analyst: Madeleine Dangazyan
Sample Type: 1.0 L Bottle-Vac™(s)
Test Notes:

Date(s) Collected: 11/2/15
Date Received: 11/5/15
Date Analyzed: 11/13/15

Client Sample ID	ALS Sample ID	Injection Volume ml(s)	Canister Dilution Factor	**Result µg/m³**	MRL µg/m³	MDL µg/m³	**Result ppbV**	MRL ppbV	MDL ppbV	Data Qualifier
VA5432	P1504757-001	1.0	1.61	6.2	6.2	2.2	0.81	0.81	0.29	**U**
VA5433	P1504757-002	1.0	1.57	**11**	6.0	2.2	**1.4**	0.79	0.28	
VA5434	P1504757-003	1.0	1.60	6.1	6.1	2.2	0.80	0.80	0.29	**U**
Method Blank	P151113-MB	1.0	1.00	3.8	3.8	1.4	0.50	0.50	0.18	**U**

U = Compound was analyzed for, but not detected above the laboratory reporting limit.
MRL = Method Reporting Limit - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client:

Client Sample ID: **Lab Control Sample**
Client Project ID: **Kirtland AFB / 140705**

ALS Project ID: P1504757
ALS Sample ID: P151113-LCS

Test Code: CARB 422 Modified
Instrument ID: HP5890 II/GC21/ECD
Analyst: Madeleine Dangazyan
Sample Type: 1.0 L Bottle-Vac™
Test Notes:

Date Collected: NA
Date Received: NA
Date Analyzed: 11/13/15
Volume(s) Analyzed: NA ml

CAS #	Compound	Spike Amount ppbV	Result ppbV	% Recovery	ALS Acceptance Limits	Data Qualifier
106-93-4	1,2-Dibromoethane	50.0	**38.2**	**76**	70-130	

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client:

Client Sample ID: **Method Blank**
Client Project ID: **Kirtland AFB / 140705**

ALS Project ID: P1504757
ALS Sample ID: P151120-MB

Test Code: EPA Method 3C Modified
Instrument ID: HP5890 II/GC1/TCD
Analyst: Nalini Lall
Sample Type: 1.0 L Bottle-Vac™
Test Notes:

Date Collected: NA
Date Received: NA
Date Analyzed: 11/20/15
Volume(s) Analyzed: 0.10 ml(s)

CAS #	Compound	Result %, v/v	MRL %, v/v	Data Qualifier
7782-44-7	Oxygen*	0.10	0.10	U
7727-37-9	Nitrogen	0.10	0.10	U
630-08-0	Carbon Monoxide	0.10	0.10	U
74-82-8	Methane	0.10	0.10	U
124-38-9	Carbon Dioxide	0.10	0.10	U

U = Compound was analyzed for, but not detected above the laboratory reporting limit.
MRL = Method Reporting Limit - The minimum quantity of a target analyte that can be confidently determined by the referenced method.
* = The oxygen result may include argon due to coelution. Ambient air includes 0.93% argon.

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client:

Client Sample ID: **Lab Control Sample**
Client Project ID: **Kirtland AFB / 140705**

ALS Project ID: P1504757
ALS Sample ID: P151120-LCS

Test Code: EPA Method 3C Modified
Instrument ID: HP5890 II/GC1/TCD
Analyst: Nalini Lall
Sample Type: 1.0 L Bottle-Vac™
Test Notes:

Date Collected: NA
Date Received: NA
Date Analyzed: 11/20/15
Volume(s) Analyzed: NA ml(s)

CAS #	Compound	Spike Amount ppmV	**Result ppmV**	**% Recovery**	ALS Acceptance Limits	Data Qualifier
7782-44-7	Oxygen*	219,000	**219,000**	**100**	84-121	
7727-37-9	Nitrogen	781,000	**779,000**	**100**	88-122	
630-08-0	Carbon Monoxide	2,000	**2,310**	**116**	87-118	
74-82-8	Methane	1,600	**1,680**	**105**	85-116	
124-38-9	Carbon Dioxide	2,000	**2,150**	**108**	84-117	

* = The oxygen result may include argon due to coelution. Ambient air includes 0.93% argon.

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client:

Client Sample ID: **Lab Control Sample** ALS Project ID: P1504757

Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-LCS

Test Code: EPA TO-15 Modified Date Collected: NA

Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA

Analyst: Evelyn Alvarez Date Analyzed: 11/19/15

Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.125 Liter(s)

Test Notes:

CAS #	Compound	Spike Amount ppbV	Result ppbV	% Recovery	DOD Acceptance Limits	Data Qualifier
115-07-1	Propene	114	**119**	**104**	57-136	
75-71-8	Dichlorodifluoromethane (CFC 12)	38.0	**32.4**	**85**	59-128	
74-87-3	Chloromethane	96.9	**76.8**	**79**	59-132	
75-01-4	Vinyl Chloride	78.3	**63.7**	**81**	64-127	
106-99-0	1,3-Butadiene	93.2	**86.8**	**93**	66-134	
74-83-9	Bromomethane	52.0	**46.2**	**89**	63-134	
75-00-3	Chloroethane	75.8	**66.7**	**88**	63-127	
67-64-1	Acetone	454	**424**	**93**	58-128	
75-69-4	Trichlorofluoromethane	38.5	**30.2**	**78**	62-126	
75-35-4	1,1-Dichloroethene	54.5	**49.3**	**90**	61-133	
75-09-2	Methylene Chloride	63.9	**52.7**	**82**	62-115	
76-13-1	Trichlorotrifluoroethane	28.7	**24.4**	**85**	66-126	
75-15-0	Carbon Disulfide	67.5	**46.6**	**69**	57-134	
156-60-5	trans-1,2-Dichloroethene	53.0	**50.3**	**95**	67-124	
75-34-3	1,1-Dichloroethane	52.4	**47.1**	**90**	68-126	
1634-04-4	Methyl tert-Butyl Ether	59.9	**57.0**	**95**	66-126	
108-05-4	Vinyl Acetate	295	**317**	**107**	56-139	
78-93-3	2-Butanone (MEK)	74.6	**73.2**	**98**	67-130	
156-59-2	cis-1,2-Dichloroethene	55.0	**52.5**	**95**	70-121	

Laboratory Control Sample percent recovery is verified and accepted based on the on-column result.
Reported results are shown in concentration units and as a result of the calculation, may vary slightly.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client: **CB&I Federal Services**
Client Sample ID: **Lab Control Sample** ALS Project ID: P1504757
Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-LCS

Test Code: EPA TO-15 Modified Date Collected: NA
Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA
Analyst: Evelyn Alvarez Date Analyzed: 11/19/15
Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.125 Liter(s)
Test Notes:

CAS #	Compound	Spike Amount ppbV	Result ppbV	% Recovery	DOD Acceptance Limits	Data Qualifier
141-78-6	Ethyl Acetate	119	**130**	**109**	65-128	
110-54-3	n-Hexane	60.2	**58.0**	**96**	63-120	
67-66-3	Chloroform	45.9	**40.6**	**88**	68-123	
109-99-9	Tetrahydrofuran (THF)	74.6	**71.8**	**96**	64-123	
107-06-2	1,2-Dichloroethane	52.9	**50.5**	**95**	65-128	
71-55-6	1,1,1-Trichloroethane	38.5	**34.1**	**89**	68-125	
71-43-2	Benzene	70.8	**66.0**	**93**	69-119	
56-23-5	Carbon Tetrachloride	36.6	**31.1**	**85**	68-132	
110-82-7	Cyclohexane	123	**117**	**95**	70-117	
78-87-5	1,2-Dichloropropane	46.8	**43.5**	**93**	69-123	
75-27-4	Bromodichloromethane	32.6	**30.6**	**94**	72-128	
79-01-6	Trichloroethene	40.2	**34.9**	**87**	71-123	
142-82-5	n-Heptane	52.7	**51.1**	**97**	69-123	
10061-01-5	cis-1,3-Dichloropropene	45.8	**46.5**	**102**	70-128	
108-10-1	4-Methyl-2-pentanone	53.7	**57.6**	**107**	67-130	
10061-02-6	trans-1,3-Dichloropropene	46.3	**48.3**	**104**	75-133	
79-00-5	1,1,2-Trichloroethane	39.6	**37.3**	**94**	73-119	
108-88-3	Toluene	57.9	**51.0**	**88**	66-119	
591-78-6	2-Hexanone	53.7	**69.8**	**130**	62-128	**L**
124-48-1	Dibromochloromethane	25.8	**24.3**	**94**	70-130	

Laboratory Control Sample percent recovery is verified and accepted based on the on-column result.
Reported results are shown in concentration units and as a result of the calculation, may vary slightly.
L = Laboratory control sample recovery outside the specified limits, results may be biased high.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client: **CB&I Federal Services**
Client Sample ID: **Lab Control Sample** ALS Project ID: P1504757
Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-LCS

Test Code: EPA TO-15 Modified Date Collected: NA
Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA
Analyst: Evelyn Alvarez Date Analyzed: 11/19/15
Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.125 Liter(s)
Test Notes:

CAS #	Compound	Spike Amount ppbV	Result ppbV	% Recovery	DOD Acceptance Limits	Data Qualifier
106-93-4	1,2-Dibromoethane	28.4	**26.3**	**93**	74-122	
127-18-4	Tetrachloroethene	29.8	**25.3**	**85**	66-124	
108-90-7	Chlorobenzene	47.8	**41.8**	**87**	70-119	
100-41-4	Ethylbenzene	50.2	**46.8**	**93**	70-124	
179601-23-1	m,p-Xylenes	98.6	**95.0**	**96**	61-134	
75-25-2	Bromoform	22.1	**17.4**	**79**	66-139	
100-42-5	Styrene	52.2	**48.9**	**94**	73-127	
95-47-6	o-Xylene	48.4	**46.9**	**97**	67-125	
79-34-5	1,1,2,2-Tetrachloroethane	30.6	**30.3**	**99**	65-127	
108-67-8	1,3,5-Trimethylbenzene	43.5	**42.6**	**98**	67-130	
95-63-6	1,2,4-Trimethylbenzene	44.4	**45.6**	**103**	66-132	
100-44-7	Benzyl Chloride	42.5	**43.8**	**103**	50-147	
541-73-1	1,3-Dichlorobenzene	37.9	**35.9**	**95**	65-130	
106-46-7	1,4-Dichlorobenzene	34.6	**31.5**	**91**	60-131	
95-50-1	1,2-Dichlorobenzene	36.6	**36.3**	**99**	63-129	
120-82-1	1,2,4-Trichlorobenzene	31.0	**24.8**	**80**	55-142	
91-20-3	Naphthalene	41.6	**42.3**	**102**	57-138	
87-68-3	Hexachlorobutadiene	21.6	**16.1**	**75**	56-138	

Laboratory Control Sample percent recovery is verified and accepted based on the on-column result.
Reported results are shown in concentration units and as a result of the calculation, may vary slightly.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client:

Client Sample ID: **Method Blank** ALS Project ID: P1504757

Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-MB

Test Code: EPA TO-15 Modified Date Collected: NA

Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA

Analyst: Evelyn Alvarez Date Analyzed: 11/19/15

Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.40 Liter(s)

Test Notes:

Canister Dilution Factor: 1.00

CAS #	Compound	Result ppbV	LOQ ppbV	LOD ppbV	MDL ppbV	Data Qualifier
115-07-1	Propene	0.73	0.73	0.58	0.20	**U**
75-71-8	Dichlorodifluoromethane (CFC 12)	0.25	0.25	0.19	0.086	**U**
74-87-3	Chloromethane	0.61	0.61	0.48	0.18	**U**
75-01-4	Vinyl Chloride	0.49	0.49	0.39	0.17	**U**
106-99-0	1,3-Butadiene	0.57	0.57	0.47	0.25	**U**
74-83-9	Bromomethane	0.32	0.32	0.26	0.12	**U**
75-00-3	Chloroethane	0.47	0.47	0.38	0.16	**U**
67-64-1	Acetone	5.3	5.3	2.3	0.81	**U**
75-69-4	Trichlorofluoromethane	0.22	0.22	0.19	0.076	**U**
75-35-4	1,1-Dichloroethene	0.32	0.32	0.28	0.11	**U**
75-09-2	Methylene Chloride	0.36	0.36	0.32	0.12	**U**
76-13-1	Trichlorotrifluoroethane	0.16	0.16	0.14	0.055	**U**
75-15-0	Carbon Disulfide	4.0	4.0	0.34	0.12	**U**
156-60-5	trans-1,2-Dichloroethene	0.32	0.32	0.26	0.12	**U**
75-34-3	1,1-Dichloroethane	0.31	0.31	0.27	0.099	**U**
1634-04-4	Methyl tert-Butyl Ether	0.35	0.35	0.31	0.12	**U**
108-05-4	Vinyl Acetate	3.6	3.6	1.5	0.46	**U**
78-93-3	2-Butanone (MEK)	4.2	4.2	0.37	0.18	**U**
156-59-2	cis-1,2-Dichloroethene	0.32	0.32	0.28	0.10	**U**

U = Undetected: The associated data value is the limit of quantitation, adjusted by any dilution factor used in the analysis.

LOQ = Limit of Quantitation - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

Verified By:_____Date:_____

Client: **CB&I Federal Services**
Client Sample ID: **Method Blank** ALS Project ID: P1504757
Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-MB

Test Code: EPA TO-15 Modified Date Collected: NA
Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA
Analyst: Evelyn Alvarez Date Analyzed: 11/19/15
Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.40 Liter(s)
Test Notes:

Canister Dilution Factor: 1.00

CAS #	Compound	Result ppbV	LOQ ppbV	LOD ppbV	MDL ppbV	Data Qualifier
141-78-6	Ethyl Acetate	0.69	0.69	0.60	0.24	**U**
110-54-3	n-Hexane	0.35	0.35	0.31	0.11	**U**
67-66-3	Chloroform	0.26	0.26	0.23	0.087	**U**
109-99-9	Tetrahydrofuran (THF)	0.42	0.42	0.37	0.17	**U**
107-06-2	1,2-Dichloroethane	0.31	0.31	0.27	0.099	**U**
71-55-6	1,1,1-Trichloroethane	0.23	0.23	0.19	0.078	**U**
71-43-2	Benzene	0.39	0.39	0.35	0.13	**U**
56-23-5	Carbon Tetrachloride	0.20	0.20	0.18	0.060	**U**
110-82-7	Cyclohexane	0.73	0.73	0.62	0.21	**U**
78-87-5	1,2-Dichloropropane	0.27	0.27	0.24	0.087	**U**
75-27-4	Bromodichloromethane	0.19	0.19	0.16	0.056	**U**
79-01-6	Trichloroethene	0.23	0.23	0.20	0.065	**U**
142-82-5	n-Heptane	0.31	0.31	0.27	0.10	**U**
10061-01-5	cis-1,3-Dichloropropene	0.28	0.28	0.23	0.077	**U**
108-10-1	4-Methyl-2-pentanone	0.31	0.31	0.27	0.098	**U**
10061-02-6	trans-1,3-Dichloropropene	0.28	0.28	0.23	0.088	**U**
79-00-5	1,1,2-Trichloroethane	0.23	0.23	0.20	0.073	**U**
108-88-3	Toluene	0.33	0.33	0.29	0.11	**U**
591-78-6	2-Hexanone	0.31	0.31	0.27	0.098	**U**
124-48-1	Dibromochloromethane	0.15	0.15	0.13	0.047	**U**

U = Undetected: The associated data value is the limit of quantitation, adjusted by any dilution factor used in the analysis.
LOQ = Limit of Quantitation - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client: **CB&I Federal Services**
Client Sample ID: **Method Blank** ALS Project ID: P1504757
Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-MB

Test Code: EPA TO-15 Modified Date Collected: NA
Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA
Analyst: Evelyn Alvarez Date Analyzed: 11/19/15
Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.40 Liter(s)
Test Notes:

Canister Dilution Factor: 1.00

CAS #	Compound	Result ppbV	LOQ ppbV	LOD ppbV	MDL ppbV	Data Qualifier
106-93-4	1,2-Dibromoethane	0.16	0.16	0.14	0.052	**U**
127-18-4	Tetrachloroethene	0.18	0.18	0.15	0.052	**U**
108-90-7	Chlorobenzene	0.27	0.27	0.24	0.087	**U**
100-41-4	Ethylbenzene	0.29	0.29	0.25	0.092	**U**
179601-23-1	m,p-Xylenes	0.58	0.58	0.50	0.17	**U**
75-25-2	Bromoform	0.12	0.12	0.11	0.036	**U**
100-42-5	Styrene	0.29	0.29	0.26	0.088	**U**
95-47-6	o-Xylene	0.29	0.29	0.24	0.086	**U**
79-34-5	1,1,2,2-Tetrachloroethane	0.18	0.18	0.15	0.055	**U**
108-67-8	1,3,5-Trimethylbenzene	0.25	0.25	0.22	0.081	**U**
95-63-6	1,2,4-Trimethylbenzene	0.25	0.25	0.22	0.076	**U**
100-44-7	Benzyl Chloride	0.24	0.24	0.22	0.053	**U**
541-73-1	1,3-Dichlorobenzene	0.21	0.21	0.19	0.062	**U**
106-46-7	1,4-Dichlorobenzene	0.21	0.21	0.17	0.058	**U**
95-50-1	1,2-Dichlorobenzene	0.21	0.21	0.18	0.062	**U**
120-82-1	1,2,4-Trichlorobenzene	0.17	0.17	0.15	0.054	**U**
91-20-3	Naphthalene	0.24	0.24	0.21	0.086	**U**
87-68-3	Hexachlorobutadiene	0.12	0.12	0.11	0.033	**U**
1330-20-7	Xylenes, Total	0.58	0.58	0.50	0.17	**U**

U = Undetected: The associated data value is the limit of quantitation, adjusted by any dilution factor used in the analysis.
LOQ = Limit of Quantitation - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

Verified By:_____Date:_____

ATTACHMENT C

ALS ENVIRONMENTAL AND TESTAMERICA SAVANNAH QUALITY ASSURANCE MANUAL AND STANDARD OPERATING PROCEDURES

C-1. TestAmerica Quality Assurance Manual, Savannah, Georgia Laboratory (Drinking Water Analyses)

C-2. TestAmerica Standard Operating Procedures, Savannah, Georgia Laboratory (Drinking Water Analyses)

C-3. ALS Environmental Quality Assurance Manual, Simi Valley, California Laboratory (Soil Vapor Analyses)

C-4. ALS Environmental Standard Operating Procedures, Simi Valley, California Laboratory (Soil Vapor Analyses)

Quality Assurance Manual

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Quality Assurance Manual
Approval Signatures

May 12, 2016

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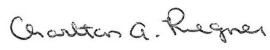

May 12, 2016

Technical Manager, Organics
Charlton Riegner

Date

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CW-E-M-001	Corporate Environmental Health and Safety Manual

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SA-AN-100	Laboratory Support Equipment (Verification and Use)
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SECTION 3. INTRODUCTION, SCOPE, AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Savannah's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- ANSI/ASQC E4-1994: "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA: 240/B-01/002. May 31, 2006)
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015..*
- *U.S. Department of Defense, Quality Systems Manual (QSM) for Environmental Laboratories, Version 4.2, October 2010.*
- *U.S. Department of Defense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013,*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, and on-line Editions.

- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP),* Version 5

3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control samples. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge, and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in the Methods Listing housed in the laboratory's information management system (i.e., TALS). The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and/or the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the

CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control and Updating procedures (refer to SOP SA-QA-001).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Savannah is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President and Chief Executive Officer (CEO), Chief Operating Officer (COO), Executive Vice President (VP) Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate and TestAmerica Savannah is presented in Figure 4-1.

4.2 Roles and Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Savannah laboratory.

4.2.2 President and Chief Executive Officer (CEO)

The President and CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. The President and CEO establishes the overall quality standard and data integrity program for the Analytical Business, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.3 Chief Operation Officer (COO)

The COO reports directly to the President and CEO of TestAmerica. The COO oversees the operations of all TestAmerica laboratories and the EMLab P&K business unit. The VPs of Operations report directly to COO.

4.2.4 Vice President of Operations

Each VP of Operations reports directly to the Executive VP of Operations and is a part of the Executive Committee. Each VP of Operations is responsible for the overall administrative and operational management of their respective laboratories. The VP's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the

financial, business, and quality objectives of TestAmerica. The VPs ensure timely compliance with Corporate Management directives, policies, and management systems reviews. The VPs are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.5 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to the President and CEO. With the aid of the Executive Committee, Laboratory Directors, Quality Directors, Safety Manager, EH&S Coordinators and QA Managers, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and EH&S Programs within TestAmerica. Additional responsibilities include:

- Review of QA/QC and EHS aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the analytical laboratories and a summary of any quality related initiatives and issues.
- Preparation of a monthly report that includes EH&S metrics across the analytical laboratories and a summary of any EH&S related initiatives and issues.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

4.2.6 Vice President of Client Service

The VP of Client Services leads the Client Service Organization (CSO) and is responsible for client satisfaction, driving operational excellence and improving client responsiveness. The VP provides direction to the Client Service Directors, Programs Managers and Project Managers.

4.2.7 Quality Assessment Director

The Quality Assessment Director reports to the VP-QA/EHS. The Quality Assessment Director has QA oversight of laboratories; is responsible for the internal audit system, schedule and procedure; monitors laboratory internal audit findings; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Compliance Director, the Quality Systems Director, and the VP-QA/EHS, the Quality Assessment Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.8 Quality Compliance Director

The Quality Compliance Director reports to the VP-QA/EHS. The Quality Compliance Director has QA oversight of laboratories; monitors and communicates DoD / DoE requirements; develops corporate tools for ensuring and improving compliance; develops corporate assessment tools; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Systems Director and the VP-

QA/EHS, the Quality Compliance Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.9 Quality Systems Director

The Quality Systems Director reports to the VP-QA/EHS. The Quality Systems Director has QA oversight of laboratories; develops quality policies, procedures and management tools; monitors and communicates regulatory and certification requirements; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Compliance Director and the VP-QA/EHS, the Quality Systems Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.10 Quality Information Manager

The Quality Information Manager is responsible for managing all company official documents (e.g., Policies, Procedures, Work Instructions), the company's accreditation database, intranet websites, external laboratory subcontracting, regulatory limits for clients on the company's TotalAccess website; internal and external client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions. The Quality Information Manager reports to the VP-QA/EHS; and works alongside the Quality Assessment, Quality Compliance and Quality System Directors and EHS Managers to support both the Analytical Quality Assurance and EHS Programs within TestAmerica.

4.2.11 Technical Services Director

The Technical Services Director is responsible for establishing, implementing and communicating TestAmerica's Analytical Business's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

4.2.12 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – Corporate Counsel & VP of Human Resources and the VP-QA/EHS. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President and CEO, VPOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and

processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.13 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

4.2.14 Environmental Health and Safety Managers (Corporate)

The EHS Managers report directly to the VP-QA/EHS. The EHS Managers are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.15 Laboratory Director

The Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective VPO. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program. Specific responsibilities include, but are not limited to, the following:

- Provides one or more Technical Managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.

- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Manager(s), and the Operations Manager as direct reports.

4.2.16 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation and improvement of the quality system based on ISO/IEC 17025, DOD ELAP, and TNI. The QA Manager is independent of production; reports directly to the Laboratory Director and their Corporate Quality Director; and has access to Corporate QA for advice and resources. The QA Manager is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. The QA Manager directs the activities of the QA Department to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities. Ensuring all personnel understand their contributions to the Quality System.
- Having documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintaining records of all ethics-related training, including the type and proof of attendance.

- Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating the document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Reviewing a percentage of all final data reports for consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, format, holding time, sensibility, and completeness of the project file contents.
- Reviewing of external audit reports and data validation requests.
- Following-up with audits to ensure client QAPP requirements are met.
- Establishing of reporting schedule and preparation of various quality reports for the Laboratory Director, clients, and/or Corporate QA.
- Developing of suggestions and recommendations to improve quality systems.
- Researching current state and federal requirements and guidelines.
- Managing the QA team to enable communication and to distribute duties and responsibilities.
- Evaluating of the thoroughness and effectiveness of training.
- Ensuring compliance with ISO/IEC 17025, DOD ELAP, and TNI.

4.2.17 Technical Manager / Director

The Technical Manager(s) report(s) directly to the Laboratory Director. The Technical Manager is accountable for all analyses and analysts under their experienced supervision and for compliance with ISO 17025, DOD ELAP, and TNI. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods (i.e., SOPs) with regard to quality, integrity, regulatory requirements, and optimum and efficient production techniques; and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He ensures that the SOPs are properly managed and adhered to at the bench.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, and the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any

deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.

- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting project QAPPs, ensuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting, and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved TALS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," ensuring that no time is lost in locating samples.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Ensuring compliance with ISO/IEC 17025, DOD ELAP, and TNI.

4.2.18 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He reports directly to the Laboratory Director. He assists the Technical Manager in determining the most efficient instrument utilization**.** Specific responsibilities include, but are not limited to, the following:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Manager and QA Manager and in compliance with regulatory requirements.
- Works with the Preventive Maintenance Coordinator to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system.

4.2.19 Compliance Officer / Environmental Health and Safety Coordinator

The Environmental Health and Safety Coordinator reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. Specific responsibilities include, but are not limited to, the following:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.

- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

4.2.20 Department Manager ~~Supervisor~~

Supervisors report to the Operations Manager. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 8.1), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, investigation of non-conformance issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.

- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.21 Analyst

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.22 Manager of Project Managers (MPM)

Specific responsibilities include, but are not limited to, the following:

- Coordinates marketing efforts with General Manager, Laboratory Director, Project Managers, and laboratory marketing group
- Supervises Project Managers
- Coordinates proposal and contract review and response process
- Responds to client inquiries

4.2.1.10 Project Manager (PM)

The PM reports to the Manager of Project Management (MPM) and serves as the interface between the laboratory's technical departments and the laboratory's clients. There is an entire staff of Project Managers that makes up the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.

- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

4.2.1.11 Custody Supervisor

The Custody Supervisor Manager reports to the Laboratory Director. He is responsible for ensuring the timely and correct shipment of sample containers, including proper preservatives and instructions, to clients. He maintains accurate records of sample container shipments. In addition, he:

- Schedules bottle orders and supervises bottle prep staff
- Supervises sample custody staff
- Coordinates with Project Managers and Field/Sampling Supervisor on scheduling field sampling efforts
- Identifies and documents custody discrepancies and notifies Project Managers about custody problems

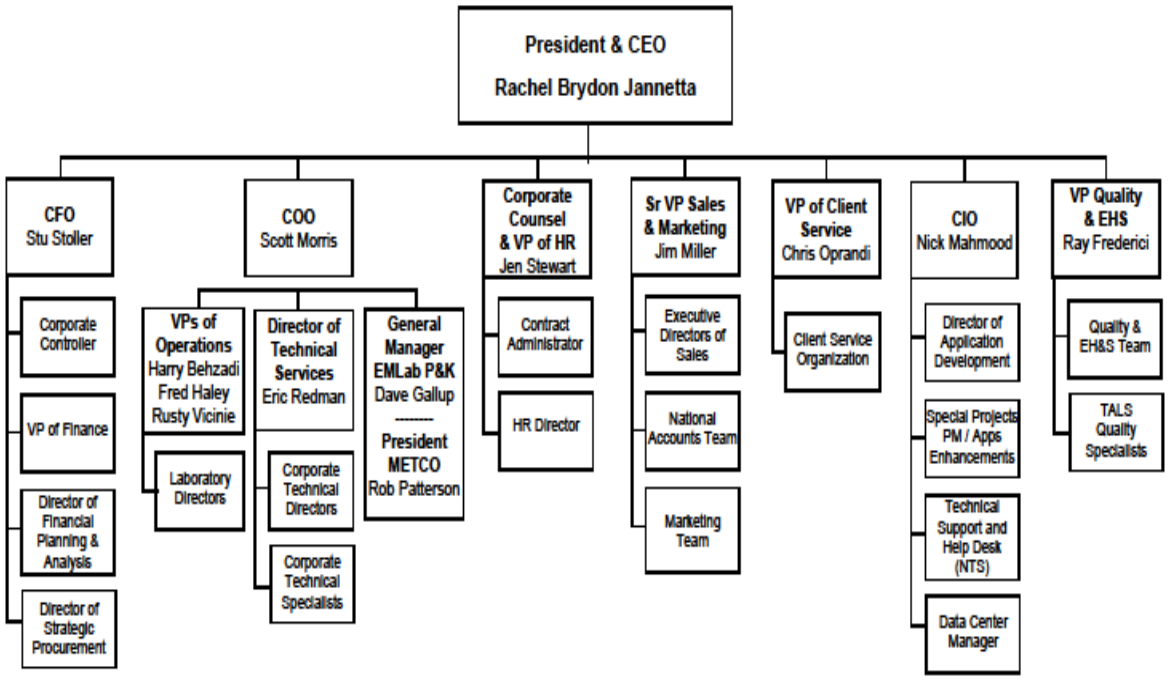
4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Laboratory Director	QA Manager
QA Manager	Laboratory Director
Operations Manager	Laboratory Director
Technical Director/Manager	Laboratory Director
EHS Coordinator	QA Manager

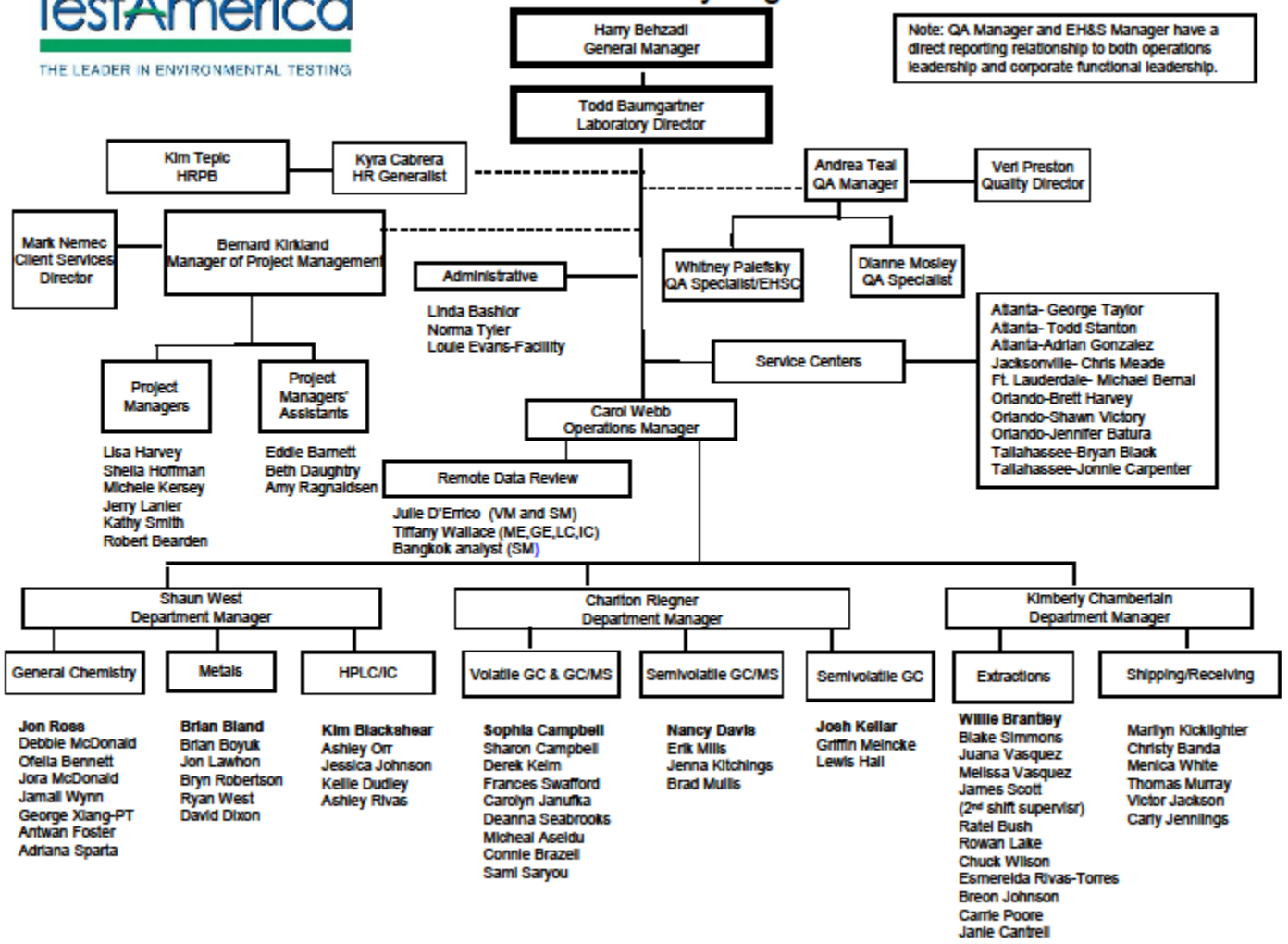
Figure 4-1. Corporate and Laboratory Organization Charts

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Savannah Laboratory Organization



SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- Comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard, and the DOD QSM, and to continually improve the effectiveness of the quality management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics and Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002)
- Procedures and guidance for recalling data if necessary. (Corporate SOP No. CA-Q-S-005)
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).

- Production of results, which are accurate and include QA/QC information that meet client pre-defined Data Quality Objectives (DQOs).
- Presentation of services in a confidential, honest, and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all laboratories. The policies described therein are typically incorporated into laboratory-specific SOPs, or the Corporate documents may be are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPPs) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or matrix spikes (MS). A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and

field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc.

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (e.g., Method Detection Limit) or quantified (e.g., Reporting Limit).

5.5 Criteria for Quality Indicators

The laboratory maintains Method Limit Groups in TALS that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA Department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP SA-QA-017: *Evaluation of Batch QC Data*.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory that are entered into the Laboratory Information Management System (i.e. TALS). The Quality Assurance Department maintains an archive of all limits used within the laboratory and stores these values in TALS. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the laboratory develops such limits from recent data in the QC database of TALS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the TALS analyte database. As sample results and the related QC are entered into TALS, the sample QC values are compared with the limits in TALS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (recommended annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.* The QA Manager evaluates control charts to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

5.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOPs)
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers, and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, *Corporate Document Control and Archiving*. The laboratory's internal document control procedure is defined in SOP SA-QA-001: *Document Control Program*.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, an employee submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System policies and procedures will be reviewed at a minimum of every year and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP refer to SOP SA-QA-001: *Document Control Program.*

Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA Department. Electronic controlled copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, refer to SOP SA-QA-001: *Document Control Program.*

Electronic copies of current documents (including QA Manuals, SOPs, Forms, Work Instructions, etc.) are maintained by the QA Department and distributed electronically via the TALS File System Shares.

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, for hardcopy distribution, obsolete documents are collected from employees according to distribution lists and are destroyed. At least one copy of the obsolete document is archived according to SOP SA-QA-001: *Document Control Program.*

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the laboratory's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals, and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel, and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the laboratory to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements, and that the lab has the capacity to meet the clients turn around needs.

For new, complex or large projects, the proposed contract is given to the Sale Directors, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002: *Contract Compliance Policy.*

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above.

The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the designated personnel is not available to review the contract, his back-up will fulfill the review requirements.

The Contracts Department maintains copies of all signed contracts.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are maintained by the Proposal Coordinator.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA Department involvement may be needed to assist in the evaluation of custom QC requirements.

PMs are the primary client contact and they ensure resources are available to meet project requirements. Although PMs do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each Project in TALS as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, email, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the supervisor.

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO/IEC 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers, Operations Managers, Department Managers, and the QA Manager are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develop laboratory and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOPs on subcontracting procedures (i.e., CA-L-S-002) and the worksharing process (i.e., CA-C-S-001).

When outsourcing analytical services, the laboratory will ensure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI and ISO/IEC 17025, and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs), Client Service Managers (CSM), or Account Executives (AE) for the export lab (i.e., the TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing, and, when possible, approval from the client shall be retained in the project folder. Standard TestAmerica Terms & Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., certain USACE projects) may require notification prior to placing such work.

8.2 Qualifying and Monitoring Subcontractors

Whenever a PM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task. Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an email from the client in the project folder.

- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract laboratory. Verify necessary accreditation, where applicable, (e.g., on the subcontractor's TNI, A2LA accreditation, or State Certification).
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned, and/or minority-owned businesses;
- TNI or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for worksharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an email is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs as specified in Corporate SOP (CA-C-S-001: *Worksharing Process*).

When the potential subcontract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager or PM begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002: *Subcontracting Procedures*.

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. Once all documents are reviewed for completeness, the Corporate QIM will forward the documents to the Purchasing Manager for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Corporate Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the

subcontracted laboratories.

- Subcontractors in good standing will be retained on the intranet listing. CSO personnel will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all CSO personnel, Laboratory Directors, QA Managers, and Sales personnel.

8.3 Oversight and Reporting

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and Corporate Counsel can tailor the document or assist with negotiations, if needed. The PM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it is current and scope-inclusive. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for all samples workshared within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor laboratory. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which laboratory produced the data for which methods and samples.

Note: The results submitted by a TestAmerica worksharing laboratory may be transferred electronically and the results reported by the TestAmerica worksharing laboratory are identified on the final report. The report must explicitly indicate which lab produced the data for which

methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFPs) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFPs allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards, and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent and Acid Lot Testing and Approval, SOP No. CA-Q-S-001. Approval information for the solvents and acids tested under CA-Q-S-001 is stored on the TestAmerica SharePoint under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

9.3.2 Receiving

It is the responsibility of the Shipping and Receiving Department to receive the shipment. Once the materials are received, the laboratory compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date to the information present on the order log.

Materials may not be released for use in the laboratory until they have been inspected and verified as suitable for use. The laboratory verifies the lot numbers of received solvents and acids against the pre-approved lists. If a material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained in TALS.

Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration date noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer or SOP expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date cannot be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of

gas should be replaced when it drops to approximately 500psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and Technical Manager must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots may be verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained electronically.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed, and Purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and it is added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate for the specific intended application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs) if a new method, and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department. Software certificates supplied by the vendors are filed with the TALS Administrator. The manufacturer's operation manual is retained electronically.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP SA-AN-100: *Laboratory Support Equipment*. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed and filed. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers and weight sets are obtained from vendors with current and valid ISO/IES 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department and filed. The equipment is then returned to service within the laboratory.

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal/bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts, or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc. As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors.

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a JD Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or Technical Management are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling process to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations, and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing, and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints, or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SA-QA-005: *Preventive and Corrective Action.*

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP SA-QA-005.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing, and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the laboratory and Quality Director in the QA Monthly Report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies, and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the Department Manager for resolution. The Department Manager may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client requests that a special procedure be applied to a sample that is not standard laboratory practice. Based on a technical evaluation, the laboratory may accept or reject the request based on technical or ethical merit. Such a request would need to be approved by laboratory management and documented in the project files. Deviations to standard operating procedures must be noted in the final report.

11.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to a member of Senior Management within 24 hours. The Senior Management staff is comprised of the Laboratory Director, Operations Manager, QA Manager, and the Technical Manager. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of Operations, and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

Corporate SOP entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-Q-S-005.

11.4 Prevention of NonConforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required, and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may

not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target, or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be provided by the laboratory to the appropriate member of Corporate QA, which serves as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing, or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will not generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, or determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval may be given by final signature on the completed corrective action report.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using NonConformance Memos (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends

- Excessive revised reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a event requiring action, the event must be defined and documented. A CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with the problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager, Operations Manager, and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each CAR is entered into a database for tracking purposes.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis. Refer to Figure 12-1.
- The QA Manager reviews monthly NCRs and CARs for trends. Highlights are included in the QA Monthly Report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, SOP SA-QA-017: *Evaluation of Batch QC Data* and the analytical SOPs.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA Monthly Report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1.
Corrective Action Report

Section 1 **Summary of Problem / Finding**
Finding #: Summary: Date Due to Agency:
Section 2 **Initial Investigation Summary**
Investigation Question #1: • Is this issue chronic (i.e., were multiple instances cited, or is the potential for similar issues present), or acute (i.e., an isolated, anomalous, or non-routine occurrence)? Response:
Investigation Question #2: • Are other departments likely to be impacted? Response:
Investigation Question #3: • Can the root cause be readily established/addressed and action items identified without further inquiry, or is further action needed to perform a formal RCA Investigation and/or develop the Corrective Action Plan? Note: If the root cause can be readily established/addressed and action items identified without further inquiry, then Section 3 does not need to be completed provided additional details are included in response to Investigation Question #4, below. Response:
Investigation Question #4: • Are there any additional comments worth noting? If so, please include. Response:
Section 3 **Root Cause Analysis Summary**
RCA Investigation Lead: RCA Investigation Team Members, if applicable:

RCA Question #1:

- Why was this finding cited?

Options:
1. Procedure/policy does not exist, is not adequate, or is not accurate.
2. Procedure/policy is in place, adequate, and accurate; however, employee did not comply.
3. Other

Response:

RCA Question #2:

- What are some underlying causes for the conclusion drawn in RCA Question #1 (i.e., what are some Quality System weaknesses indicated by this issue that also need to be addressed)?

Note: There may be more than one underlying cause/weakness, and each underlying cause/weakness may in turn have other underlying causes/weaknesses.

Examples:
1. Insufficient or incomplete method validation procedures.
2. Trend analysis was not performed or is insufficient.
3. Insufficient or incorrect detail in SOPs; SOPs out of date; SOPs do not match current practice, etc.
4. Missing or inadequate mechanism to capture information (e.g., form, spreadsheet, Data Types, etc.).
5. Missing or inadequate training.
6. Insufficient employee oversight / supervision.
7. Ineffective primary data review process.
8. Ineffective self-monitoring process (e.g., notebook review, secondary data review, internal audits, etc.).
9. Personnel problem, insufficient resources, lack of attention to detail, etc.
10. Insufficient reagent traceability or control procedures.
11. Poor communication channels.
12. Improper or inadequate equipment maintenance procedures.
13. Ineffective Document Control mechanisms
14. Ineffective sample scheduling mechanisms, workflow, backlogs, etc.
15. Other

Response:

RCA Question #3:

- Is a Data Recall, an SOP revision, or additional training needed?

Response:

RCA Question #4:

- Are there any additional comments worth noting? If so, please include.

Response:

Section 4
Corrective Action Assignments

<<Based on the Initial Investigation and/or Root Cause Analysis Summary outlined above, what action items are needed to: 1) correct the original finding, and 2) minimize its recurrence? >>

Action Item #1:

Assigned Party:
Due Date:
Status:

Actions Taken:

Supporting Documentation Attached:

Section 5
Audit Response Documentation

Laboratory Response sent to agency on: XXXXX, attached here.

Section 6
Subsequent Information / Documentation Requests from Agency

Summary:

Assigned to:
Due Date:

Documentation attached here:

Section 7
Additional Close-Out / Follow-Up and Comments

A)
This finding pertains to an isolated and/or anomalous event. The corrective action taken is sufficient to address this issue. No further action or follow-up is needed at this time to close out this item.
Initial / Date:

B)
An additional routine follow-up assessment is required to evaluate the effectiveness of the corrective action taken.
Follow-up Assigned To:
Due Date:
Documentation Needed:
Items used to assess effectiveness/sustainability of corrective action:
<<Include AD batch numbers, attach example logbook pages, etc., as applicable.>>

Choose One:
a) Corrective action has been implemented and is effective.
b) Similar problems have been noted. The corrective action has not been effective. Additional action is required.
Initial / Date:

Table 12-1. General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Instrument Blank *(Analyst)*	- Criteria in analytical SOP	- Prepare and analyze another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Analyst)*	- Criteria in analytical SOP	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Initial Calibration Verification (Second Source ICV) *(Analyst)*	- Criteria within analytical SOP	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Verification (CCV) *(Analyst)*	- Criteria within analytical SOP	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst)*	- Criteria in TALS MLGs	- If matrix interferences are present, evaluate the LCS. - If the LCS is within acceptable limits the batch is acceptable.
Laboratory Control Sample (LCS) *(Analyst)*	- Criteria in TALS MLGs and SOP SA-QA-017	- Reanalyze LCS. - Batch must be re-prepared and/or re-analyzed.
Surrogates *(Analyst)*	- Criteria in TALS MLGs	- Individual sample must be repeated, unless obvious matrix interference is noted.
Method Blank *(Analyst)*	<1/2RL	- Reanalyze blank. - Determine source of contamination. - Re-prepare/re-analyze batch.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems, and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report
- trending NCMs
- review of control charts and QC results
- trending proficiency testing (PT) results
- performance of management system reviews
- trending client complaints
- review of processing operations
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly be the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lessons Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.

- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions/Process Improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance, and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA Department on the Q-Drive and are backed up as part of the regular network backup. Technical records are maintained by the laboratory departments in the Data Archival folder on the Public_QA Drive and are backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer- or hand-generated (some records may be in both formats).

Table 14-1. Records Index[1]

	Record Types [1]:	Retention Time:
Technical Records	Raw Data Logbooks[2] Standards Certificates Analytical Records MDLs/IDLs/DOCs Lab Reports	5 Years from analytical report issue*
Official Documents	Quality Assurance Manual (QAM) Work Instructions Policies SOPs	5 Years from document retirement date*
QA Records	Internal & External Audits/Responses Certifications Corrective/Preventive Actions Management Reviews Method & Software Validation Data Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	Sample Receipt & COC Documentation Contracts and Amendments Correspondence QAPP SAP Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual and Permits	5 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies Technical Training Records	Refer to HR Manual

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance Log, Instrument Run Log, Preparation Logs (standard and samples), Standard and Reagent Receipt Logs, Balance Calibrations, Temperature Logs, etc.
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2. Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	5 years (project records) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio VAP	10 years and State contacted prior to disposal

Program	[1]Retention Requirement
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 for more information. Electronic records are maintained in the Data Archival Folder on the Public_QA drive, or in another applicable drive (such as Q-drive). Refer to SOP SA-QA-001: *Document Control Program* for specific information on the archival, storage, and back-up of records.

14.1.4 The recordkeeping system allows for historical reconstruction of all laboratory activities that produced the analytical data as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The chain of custody would indicate the name of the sampler.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The recordkeeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set, etc. as per SOP SA-QA-001: *Document Control Program).* Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Where an analysis is performed without an instrument, TALS sheets, bound logbooks, bench sheets, or spreadsheets are used to record and file data. Standard and reagent information is recorded in TALS for each method.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning

process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

14.2 Technical and Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records, and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code**;**
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the TALS and on specific analytical report formats.

14.2.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a documented supervisory or peer review on a monthly basis.

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates, and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the TALS. Records are considered archived when noted as such in the records management system.

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation, or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and**,** when requested**,** to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits / QA Technical Audits	Joint Responsibility: a) QA Manager or designee, b) Technical Manager or designee (Refer to SOP No. CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint Responsibility: a) QA Manager or designee, b) Technical Manager or designee (Refer to SOP No. CW-Q-S-003)	SOP Compliance Review Frequency: - Every 2 years (non-DOD SOPs) - 100% of SOPs annually (DOD SOPs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness and sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., CHROM AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period. All analysts should be reviewed over the course of a two year period through at least one QA Technical Audit.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every year. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities (new IDOC), reviews of the analyst work products will be performed.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

Single blind performance audits are employed for several reasons. One purpose is to provide corrective action for parameters judged to be unacceptable on external or internal performance audits. Periodic internal performance audits are also used to test parameters that are not routinely tested by external performance audits. Finally, single blind performance audits are employed to satisfy certain certification requirements, to satisfy auditors' specific requests for performance audit samples, or to provide additional evidence of data quality to clients with specific questions regarding laboratory performance.

The laboratory participates semi-annually in performance audits conducted through the analysis of Proficiency Testing (PT) samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-Potable Water, and Soil.

These PT studies are performed approximately six months apart. The first study of the year is usually performed in January, and the second study is usually performed in July. The PT results are submitted to certification agencies directly from the PT Provider. Remedial PT studies can be performed, as required, for any analytes scored as unacceptable. Root cause investigation into any unacceptable results must be initiated. Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the laboratory's corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible

laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department and/or Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the laboratory's corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Quality Director, and the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and VPs of Operations.

16.2 Annual Management Review

The senior laboratory management team (Laboratory Director, Operations Manager, QA Manager) conducts a review annually of its quality systems and TALS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives, and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel can be included in this meeting at the discretion of the Laboratory Director. The TALS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the TALS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
- Adequacy of staff, equipment and facility resources.

- Adequacy of policies and procedures.
- Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate VP of Operations and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table).

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Internal Investigations SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's President and CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations, and Quality Directors receive a monthly report from the VP-QA/EHS summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific laboratories.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures, and records management.

Laboratory management is responsible for formulating goals for laboratory staff with respect to education, training, and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the laboratory staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform, or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources webpage. (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Director – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

Specialty	Education	Experience
Technical Director - Microbiology	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics (New Hires)	1 week of hire	All
Ethics (Comprehensive)	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics (Comprehensive Refresher)	Annually	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood, and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques, or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst's knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicating to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the SOP SA-QA-006: *Training Procedures*.

17.4 Data Integrity and Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive ethics training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 55,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control, and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and TALS are regulated to protect against raw data loss.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas available to ensure unencumbered work. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys and alarm codes are distributed to employees as necessary.

Employees wear photographic identification name cards while on the premises.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage, and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOP)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints, as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP SA-QA-001: *Document Control*.
- SOPs are reviewed at a minimum of annually, and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*

- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008, Final Update V, August 2015.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method, or personnel (e.g., analyst has not performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve, and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented, and the laboratory informs the client of its procedure for working with unusual compounds.

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Refer to SOP SA-QA-006: *Training Procedures* for information on performing Initial Demonstrations of Capability (IDOC).

A certification statement (refer to Figure 19-1) can be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper

quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of method performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks, or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value can be differentiated from blanks. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)* for details on the laboratory's MDL process.

19.8 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of instrument blanks and calculating three times the absolute value of the standard deviation.

If IDL is greater than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP SA-QA-008 for further details.

The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.11 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, and chromatography retention time windows.

19.12 Estimation of Uncertainty of Measurement

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty" (i.e., the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2).

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5mg/L.

Refer to SOP SA-QA-017: *Evaluation of Batch QC Data* for more information on this topic.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., EPA 524.2, EPA 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also

variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client-specific, contractual Terms and Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the laboratory was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to non-homogenous samples, Encores/Terracores, and sodium bisulfate preserved samples.

19.14 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the TestAmerica LIMS System (TALS) which is a custom in-house developed TALS system that has been highly customized to meet the needs of the laboratory. It is referred to as TALS for the remainder of this section. TALS utilizes Microsoft SQL Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 Maintain the Database Integrity: Assurance that data are reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal TALS permissions procedure.

- TALS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails, and controlled access.

19.14.1.2 **Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 **Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to approving the data in TALS.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and SOP SA-QA-008: *Evaluation of Chromatographic Data.*

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data is retained in the laboratory benchsheets, computer file (if appropriate), and/or runlog. All criteria pertinent to the method are recorded. The documentation is recorded at the time observations or calculations are made and each person involved is readily identified.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter (ug/L) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (ug/kg) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%. Units are defined in each laboratory SOP.

19.14.2.3 In general, results are reported to 2 significant figures on the final report.

19.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the TALS, the raw results and dilution factors are entered directly into TALS by the analyst, and the software calculates the final result for the analytical report.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with

the TALS, the raw results and dilution factors are transferred into TALS electronically. Electronic data from instruments are saved electronically in a daily folder on the system (CHROM or instrument computer). For instruments that print out calibrations and concentrations, the data are retained with the data file. The data file is stored in the Archival Folder on the Public_QA. These files are transferred to the server daily, and eventually, to a tape file.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Department controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Data review procedures are outlined in the analytical SOPs and SOP SA-QA-002: *Data Generation and Review* and ensure that data reported are free from calculation and transcription errors and that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing manual integrations to ensure the authenticity of the data (SOP SA-QA-008). The general review concepts are discussed below; more specific information can be found in the SOPs.

19.14.4.1 Log-in Review - The data review process at starts at the sample receipt stage. Sample Control personnel review chain-of-custody forms and input the sample information into the TALS. The Project Management Assistant reviews the transaction of the chain-of-custody forms and inputs the required analyses. The Project Managers perform final review of the chain-of-custody forms and entered information.

19.14.4.2 First Level Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analysts transfer the data into TALS and data qualifiers are added as needed. All first level reviews are documented.

19.14.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data

(e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples have unusually high results
- Samples exceed a known regulatory limit
- Raw data indicates some type of contamination or poor technique
- Inconsistent peak integration is observed
- Transcription errors are identified
- Results are outside of calibration range

19.14.4.4 Unacceptable analytical results may require reanalysis of the samples. Problems may be brought to the attention of the Laboratory Director, Project Manager, Operations Manager, Quality Assurance Director/Manager, Technical Manager, or Department Manager for further investigation, if needed. Corrective action is initiated whenever necessary.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/narratives are present, data qualifiers are appropriate, and project-specific requirements are met. The following are some examples of chemical relationships that can be reviewed (if data is available):

- Total Results are $\geq$ Dissolved results (e.g. metals)
- Total Solids (TS) $\geq$ Total Dissolved Solids (TDS) or Total Suspended Solids (TSS)
- TKN $\geq$ Ammonia
- Total Phosphorus $\geq$ Orthophosphate
- COD $\geq$ TOC
- Total Cyanide $\geq$ Amenable Cyanide
- TDS $\geq$ individual anions

19.14.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report and sends to the client.

19.14.4.7 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal SOP No. SA-QA-008, entitled *Evaluation of Chromatographic Data*.

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integration is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Demonstration of Capability Documentation

TRAINING DOCUMENTATION FORM
DEMONSTRATION OF CAPABILITY

Laboratory Name: TestAmerica Savannah
Address: 5102 LaRoche Avenue
Savannah, GA 31404

Date Completed: _____

Analyst Name: _____

Prep Analyst Name (s): _____

Analytical Test Method: _____

Prep Method: _____

Matrix: ☐ Soil ☐ Aqueous ☐ Other

Analytical SOP Document Control Number: _____

Prep SOP Document Control Number: _____

Analyte, Class of Analytes, or Measured Parameters: _____

If PT Study is used as DOC, list the PT Number: _____

We, the undersigned, CERTIFY that:
1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program and/or other state and federal programs have completed the Demonstration of Capability.
2. The test method(s) was performed by the analyst(s) identified on this certification.
3. A copy of test method(s) and laboratory-specific SOPs are available for all personnel on-site.
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

Technical Director's Name	Signature	Date
Quality Assurance Officer's Name	Signature	Date

FQA049:08.13.07:6

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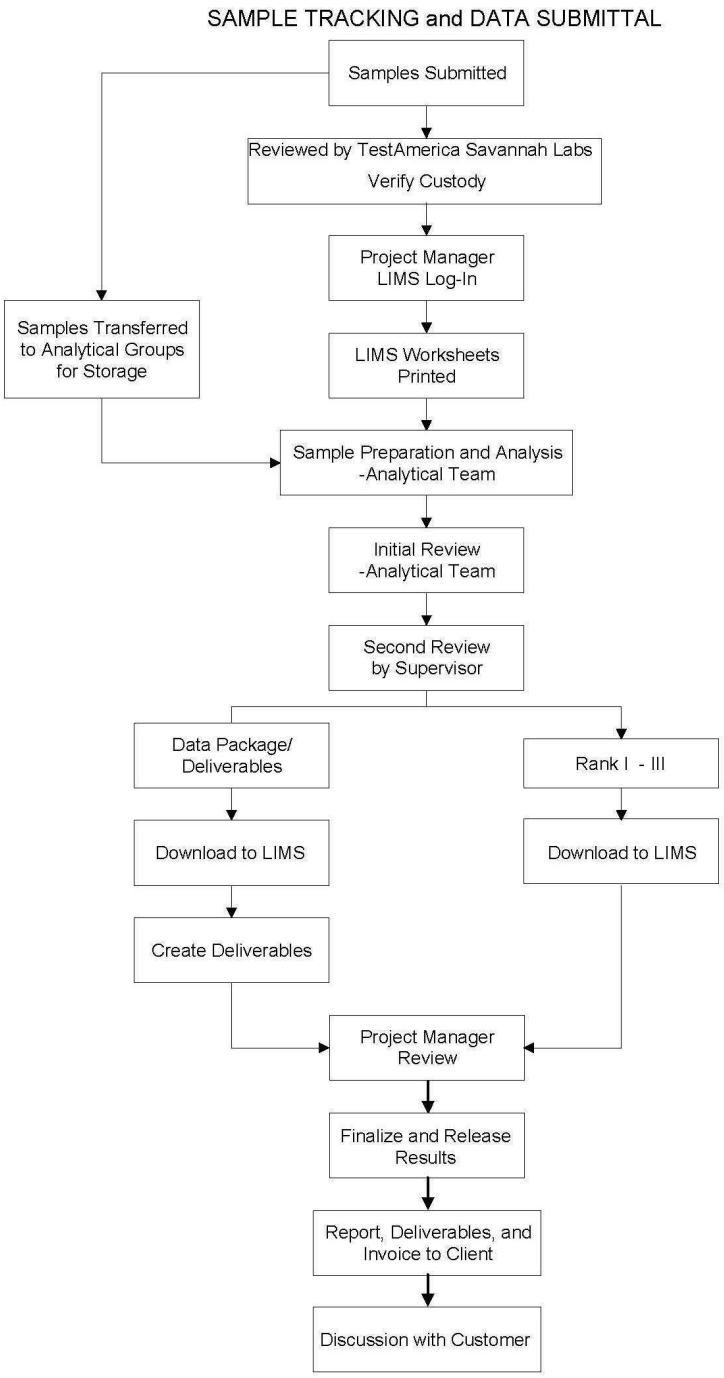
Figure 19-2. Work Flow



SECTION 20. EQUIPMENT AND CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the logbook used to monitor performance is also the maintenance logbook. Multiple pieces of equipment may share the same logbook as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logbooks are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logbooks shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control (e.g. CCV run on *'date'* was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to laboratory operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage, or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult the analytical SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer.

- If the temperature measuring device is used over a range of 10℃ or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10℃, then the verification must bracket the range of use.

IR thermometers, digital probes, and thermocouples are calibrated quarterly. IR Thermometers should be calibrated over the full range of use, including ambient and iced (4 degrees) per the EPA Drinking Water Manual.

The mercury NIST thermometer is recalibrated every three years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented electronically. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in equipment-specific logbooks or TALS sample batches. More information on this subject can be found in SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day – including weekends and holidays (i.e., 7 days a week).

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented on the Daily Temperature Log and in procedure-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Glass micro-syringes are considered the same as Class A glassware provided they are purchased with a manufacturer's certificate attesting to their accuracy. Micro-syringes are routinely purchased from Hamilton Company. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

Any device not regularly verified can not be used for any quantitative measurements.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration).

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed, if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules are standard ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or a vendor-certified different lot if a second source is not available). For unique situations, where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used, then bracketing calibration verification standards are not required, only daily verifications are needed, unless specified by the reference method. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements. Refer to the specific SOPs for requirements. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument/method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client:

a) when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b) when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated, and accepted.

Samples reported by the two conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

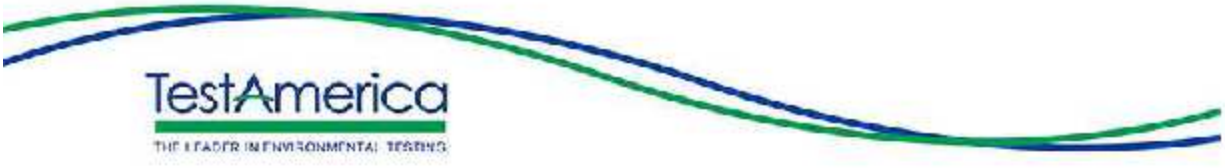
For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS Tuning

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Instrumentation



TestAmerica Savannah Instrument List

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
ICP	Varian (ICP E)	730-ES	IP0712M054	2008	New
ICP	Varian (ICP F)	730-ES	1P0803M118	2012	Used
ICP/MS	Agilent (ICP/MS B)	Agilent 7500CE G3272A	JP 14101289	2005	New
ICP/MS	Agilent (ICP/MS C)	Agilent 7700x G3281A	JP10390615	2011	New
CVAA	Leeman (2)	HYDRA AA II	00024	2011	New
GC/MS Semivolatiles	Hewlett-Packard (CMS D)	5973/6890	US82311451	1999	New
GC/MS Semivolatiles	Hewlett-Packard (CMS E)	5973/6890	US82311455	1999	New
GC/MS Semivolatiles	Hewlett-Packard (CMS F)	5973/6890	US44647039	2004	New
GC/MS Semivolatiles	Hewlett-Packard (CMS G)	5973/6890	US82311571	1999	New
GC/MS Semivolatiles	Hewlett-Packard (CMS K)	5973/6890	CN10524062	2005	New
GC/MS Semivolatiles	Hewlett-Packard CMS (N)	5973/6890	US72010580	1998	New
GC/MS Semivolatiles	Hewlett-Packard (CMS R)	5973/6890N	21842170	2002	New
GC/MS Semivolatiles	Hewlett-Packard (CMS T)	5973/6890	US33246115	2003	New
GC/MS Semivolatiles	Agilent (CMS W)	5975/6890N	US10608004	2006	New
GC/MS Semivolatiles	Hewlett-Packard (CMS X)	5975/6890N	CN10608061	2006	New
GC/MS Semivolatiles	Agilent (CMS Y)	5975/7980A	US80838915	2008	New
GC/MS Semivolatiles	Hewlett-Packard (CMSAE)	5973/6890	US82311503	2015	Used
GC/MS Volatiles	Hewlett-Packard (CMS A)	5973/6890	US82311453	2000	New
GC/MS Volatiles	Hewlett-Packard (CMS AA)	5973/6890N	US406220567	2013	Used

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GC/MS Volatiles	Hewlett-Packard (CMS B)	5973/6890	US82311452	2000	New
GC/MS Volatiles	Hewlett-Packard (CMS C)	5975/7890	CN10917056	2012	New
GC/MS Volatiles	Hewlett-Packard (CMS O)	5973/6890	US7200579	1993	New
GC/MS Volatiles	Hewlett-Packard (CMS P)	5973/6890	US0039011	2000	New
GC/MS Volatiles	Hewlett-Packard (CMS S)	5973/6890	US21843181	2002	New
GC/MS Volatiles	Agilent (CMS U)	5973/6890	US52441057	2005	New
GC/MS Volatiles	Agilent (CMSAC)	5973/6890	US82311488	2014	Transfer
GC/MS Volatiles	Agilent (CMSAD)	5973/6890	US82311485	2014	Transfer
Ion Chromatograph	Dionex (CIC N)	ICS-1000	04060105		Used
Ion Chromatograph	Dionex (CIC G)	ICS-2000	05101132	2005	New
Ion Chromatograph	Dionex (CIC H)	ICS-2000	06080799	2006	New
Ion Chromatograph	Dionex (CIC K)	ICS-2000	0307011	2012	Used
Ion Chromatograph	Dionex (CIC L)	ICS-2000	05120486	2012	Used
GC Semivolatiles	Hewlett-Packard (CSG J)	6890 (ECD)	US00033184	2000	New
GC Semivolatiles	Hewlett-Packard (CSG K)	6890 (ECD)	US10223085	2002	New
GC Semivolatiles	Agilent (CSG Q)	6890N (FID)	CN10521056	2005	New
GC Semivolatiles	Hewlett-Packard (CSG S)	6890 Plus (ECD)	US00024188	2000	New
GC Semivolatiles	Hewlett-Packard (CSG X)	6890N (ECD)	CN10406086	2003	New
GC Semivolatiles	Agilent (CSG Y)	6890N (ECD)	CN10528081	2005	New
GC Semivolatiles	Agilent (CSG Z)	6890N (ECD)	CN10814004	2008	New
GC Semivolatiles	Agilent (CSGAA)	6890 (ECD)	US00031692	2013	Used
GC Semivolatiles	Agilent (CSG AB)	6890N(FID)	US10224026	2013	Used
GC Semivolatiles	Agilent (CSG AD)	6890N (ECD)	4510326002	2015	Used
GC Volatiles	Agilent (CVG G)	6890 (FID)	14921	2007	New
GC Volatiles	Agilent (CVG U)	6890 (FID)	US10439011	2005	New

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GC Volatiles	Agilent (CVG V)	6890 (FID)	CN10619098	2006	New
GC Volatiles	Agilent (CVG W)	6890 (FID)	CN10603131	2006	New
Liquid Chromatography	Hewlett-Packard (CLC J)	1100	US72101013	2002	New
Liquid Chromatography	Hewlett-Packard (CLC K)	1100	US72102590	2002	New
Liquid Chromatography	Hewlett-Packard (CLC N)	1100	DE91607527	2008	Used
General Chemistry	Hach (TURB1)	2100 AN	950400000487	1995	New
General Chemistry	Lachat (1)	QuickChem 8000	A83000-1070	1997	New
General Chemistry	Lachat (2)	QC 8500 Series 2	100200001169	2010	New
General Chemistry	Lachat (3)	QuickChem 8000	A8300-1086	2012	Used
General Chemistry	BOD AssayPlus	Version 3.0	270F6XB334	2006	New
General Chemistry	PCTitrate	Version 3.0	270G6XB370	2006	New
General Chemistry	Konelab (1)	Konelab20	M4218134	2000	New
General Chemistry	Konelab (2)	Konelab20	M3118114	2001	New
General Chemistry	Konelab (3)	Konelab20	S2519236	2013	Used
General Chemistry	Analyik jena	Multi X 2500	N1-197/O	2014	New
General Chemistry	Shimadzu	TOC-L	H54335232053	2015	New
General Chemistry	Hach (SPC7)	DR2800	1359509	2016	Used
Extractions	Hach (SP6)	DR2800	1342967	2016	Used

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, deionized (DI) water systems, automatic pipettes, and other volumetric measuring devices. (Refer to Section 20.3.) With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices (daily for DOD). Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A glassware and glass microliter syringes should be routinely inspected for chips, acid etching, or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia-Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

A calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty. The calibration report or certificate contains a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. All calibration reports are filed in the QA Department.

An external certified service provider services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. Mercury thermometers are verified annually against a traceable reference thermometer. Digital thermometers are verified quarterly against a traceable reference thermometer. Temperature readings of ovens, refrigerators, freezers, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique reagent ID and expiration date. All documentation received with the reference standard is retained as a QC record and references the reagent ID.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be, at a minimum, the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. (Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.)

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained electronically. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's TALS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the TALS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- TALS Standard ID
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained electronically.

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container
- Recommended Storage Conditions

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets, and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

TestAmerica Savannah provides limited sampling services. Sampling procedures are described in SOP SA-FD-05: *Field Sampling Procedures.*

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness provided by the supplier are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote, or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need

consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples.*

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time, and location of sampling
- Sample collector's name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of

the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (e.g., Fed-Ex, UPS), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date as it lists all receipts on each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

Additional information on the sample receipt process is given in SOP SA-CU-01: *Sample Receipt Procedures.*

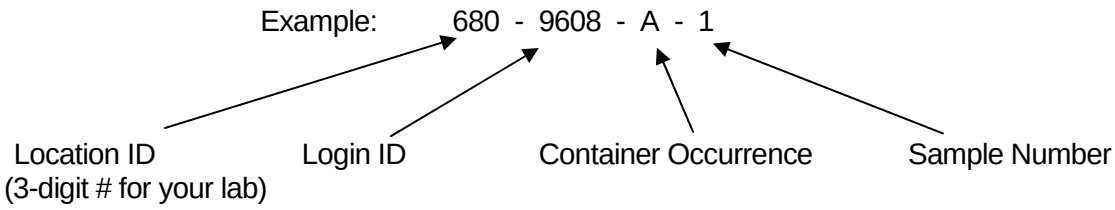
23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on the Sample Receipt Checklist in TALS and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at any time. This system includes identification for all samples, subsamples, and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Savannah is the laboratory (Location ID 680). The Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 680 - 9608 - A - 1 - **A** ←——**Secondary Container Occurrence**

Example: 680-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the Project Manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and route them to the appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

23.4 Sample Storage

In order to avoid deterioration, contamination, or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers, or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, storage blanks are maintained in the volatile sample refrigerators and analyzed every week.

Analysts retrieve the sample container allocated for their analysis from the designated storage location, prepare or analyze the sample, and return the remaining sample to the storage location from which it originally came. All samples are scanned into and out of the storage locations using the TALS sample custody program. Empty containers are scanned into the TALS sample custody program as empty and are properly disposed of. All samples are kept for at least 30 days after the report is sent out, which meets or exceeds most sample holding times. After this time, the samples are properly disposed of in accordance with the Environmental Health and Safety Manual.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples and Foreign Soils

Upon receipt, foreign soil samples are marked with a fluorescent green “FOREIGN SOIL” label prior to distributing to the analytical departments. Once the sample is received by the department, it is stored in a “FOREIGN SOIL ONLY” box segregated from other samples. Non-hazardous foreign soil samples are sent out for incineration by a USDA-approved waste

disposal facility. RCRA hazardous foreign soil samples are heat treated at the laboratory. After heat treatment, normal disposal procedures are followed. Refer to the Environmental Health and Safety Manual Addendum for additional information on disposal of hazardous samples. If not classified as hazardous, foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they did not inadvertently omit a key part of regulatory compliance testing.

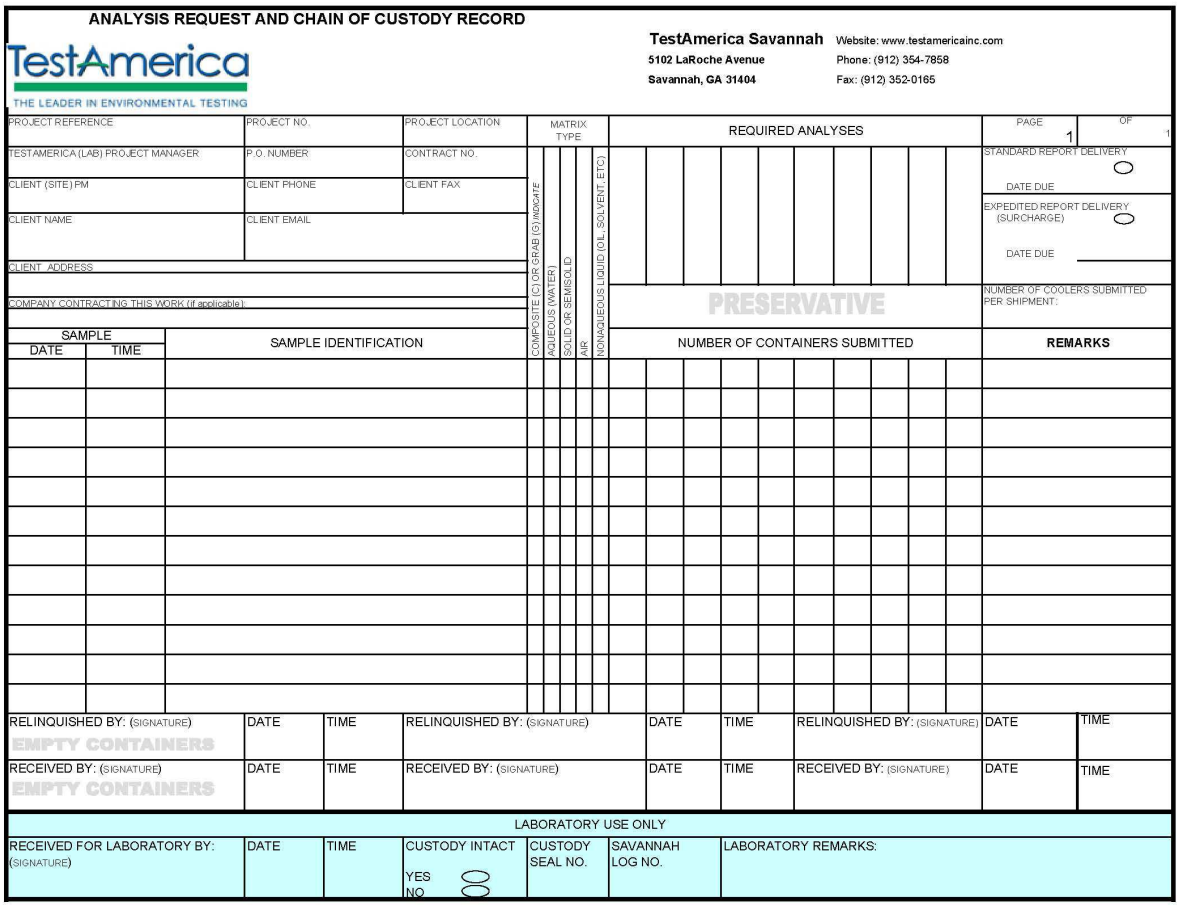
23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures outlined in the Savannah Addendum to the Environmental Health and Safety Manual. All procedures in the laboratory Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than three months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or

deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Chain of Custody (COC)



ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Savannah Website: www.testamericainc.com
5102 LaRoche Avenue Phone: (912) 354-7858
Savannah, GA 31404 Fax: (912) 352-0165

PROJECT REFERENCE	PROJECT NO.	PROJECT LOCATION
TESTAMERICA (LAB) PROJECT MANAGER	P.O. NUMBER	CONTRACT NO.
CLIENT (SITE) PM	CLIENT PHONE	CLIENT FAX
CLIENT NAME	CLIENT EMAIL	
CLIENT ADDRESS		
COMPANY CONTRACTING THIS WORK (if applicable):		

MATRIX TYPE: COMPOSITE (C) OR GRAB (G) INDICATE; AQUEOUS (WATER); SOLID OR SEMISOLID; AIR; NONAQUEOUS LIQUID (OIL, SOLVENT, ETC)

REQUIRED ANALYSES

PAGE 1 OF 1

STANDARD REPORT DELIVERY ⬭
DATE DUE
EXPEDITED REPORT DELIVERY (SURCHARGE) ⬭
DATE DUE

PRESERVATIVE

NUMBER OF COOLERS SUBMITTED PER SHIPMENT:

SAMPLE DATE	SAMPLE TIME	SAMPLE IDENTIFICATION	NUMBER OF CONTAINERS SUBMITTED	REMARKS

RELINQUISHED BY: (SIGNATURE) EMPTY CONTAINERS	DATE	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME
RECEIVED BY: (SIGNATURE) EMPTY CONTAINERS	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME

LABORATORY USE ONLY

RECEIVED FOR LABORATORY BY: (SIGNATURE)	DATE	TIME	CUSTODY INTACT YES ⬭ NO ⬭	CUSTODY SEAL NO.	SAVANNAH LOG NO.	LABORATORY REMARKS:

FCU052:01.02.08:2

Figure 23-2. Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax, or e-mail ASAP after the receipt of the samples.

1) Samples must arrive in good condition with a Chain-of-Custody (COC) filled out completely.

2) Samples must be properly labeled.

3) Samples must be in proper containers with adequate volume for the analysis. Aqueous samples submitted for Volatiles analyses must be submitted without headspace. Samples must be dechlorinated and submitted with proper chemical preservation (pH) as required by the analytical test method.

4) Most analytical methods require chilling samples to 4^{o}C. These criteria are met if the samples are chilled to below 6^{o}C and above freezing. Note: Samples hand-delivered to the laboratory immediately after collection are only considered acceptable if there is evidence that the chilling process has begun (i.e., arrival on ice).

5) Samples must be prepared and analyzed with the holding times defined in the analytical test method.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

Figure 23-3. Login Sample Receipt Checklist

Login Sample Receipt Check List

Client: TestAmerica Laboratories, Inc.

Job Number:

SDG Number:

Login Number:

List Source:

Creator:

List Number:

Question	T / F/ NA	Comment
Radioactivity either was not measured or, if measured, is at or below background		
The cooler's custody seal, if present, is intact.		
The cooler or samples do not appear to have been compromised or tampered with.		
Samples were received on ice.		
Cooler Temperature is acceptable.		
Cooler Temperature is recorded.		
COC is present.		
COC is filled out in ink and legible.		
COC is filled out with all pertinent information.		
There are no discrepancies between the sample IDs on the containers and the COC.		
Samples are received within Holding Time.		
Sample containers have legible labels.		
Containers are not broken or leaking.		
Sample collection date/times are provided.		
Appropriate sample containers are used.		
Sample bottles are completely filled.		
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs		
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.		
If necessary, staff have been informed of any short hold time or quick TAT needs		
Multiphasic samples are not present.		
Samples do not require splitting or compositing.		
Is the Field Sampler's name present on COC?		
Sample Preservation Verified		

TestAmerica Savannah

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying, and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Control Type	Details
Method Blank (MB)	Used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	Prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

Control Type	Details
Instrument Blanks	Blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	Required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	Sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	Sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Holding Blanks	Referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon method performance (e.g., Laboratory Control Sample), which entails both the preparation and measurement steps; and matrix effects (e.g., Matrix Spike or Sample Duplicate), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology, and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as phosphorus), a calibration verification standard is reported as the LCS.

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, Toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSDs are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS

and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or surrogate spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary (recommended on an annual basis) unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (CCV) unless the analytical method specifies a tighter limit.

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- For routine analytes that are not classified as poor performers, the lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable).

- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart may be visually inspected and, using professional judgment, they may be left unchanged if there is no effect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

The QA Department generates a Method Limit Group (MLG) in the TALS that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Savannah. The MLG includes an effective date and is updated each time new limits are generated and entered. Unless otherwise noted, limits within these tables are

laboratory generated. The TALS maintains an archive of all limits used within the laboratory.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- If there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

Marginal exceedances should be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is reviewed and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report) with a "Result" column header.

25.2.2 Each report cover page includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. Job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Reporting limits

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.19 When TNI accreditation is required, the laboratory shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.20 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.21 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.22 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.23 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or preliminary report). A complete report must be sent once all of the work has been completed.

25.2.24 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.25 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above, excluding QC data.
- Level II is a Level I report plus summary information, including QC results.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. TestAmerica Savannah offers a variety of EDD formats including Environmental Resources Program Information Management System (ERPIMS), Automated Data Review (ADR), Locus Focus (EIM), EQUIS ESBasic, Environmental Quality Information Systems (EQUIS), Staged Electronic Data Deliverable (SEDD), EPA Region V EDD (EDMAN), and Terrabase.

EDD specifications are submitted to the IT department by the Project Manager for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without

errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify sender immediately.

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained in the TALS, as is the original report. The revised report is stored in the project files under the sample number followed by "Rev#" where # is the number of the report revision.

When the report is re-issued, the revision number is placed on the cover/signature page of the report or at the top of the narrative page. A brief explanation of reason for the re-issue and a reference back to the last final report generated may be included.

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

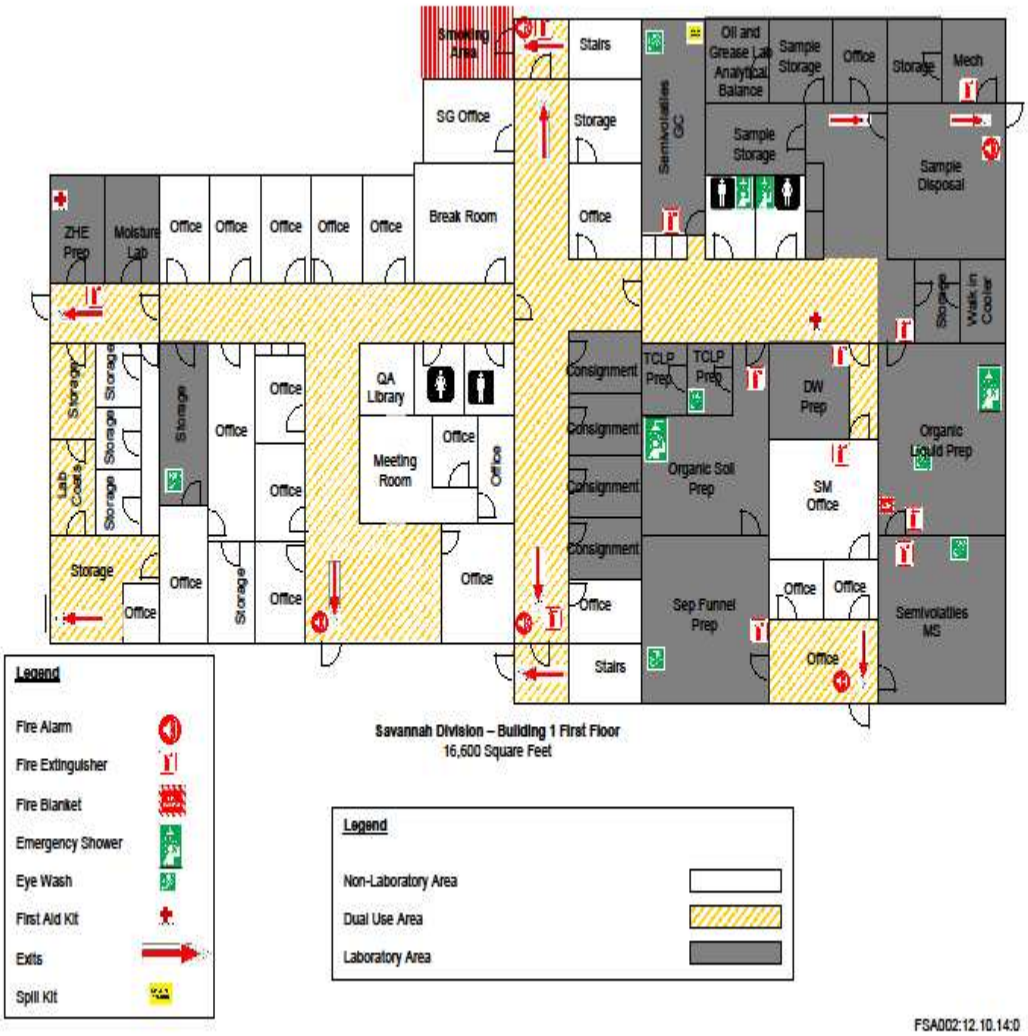
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

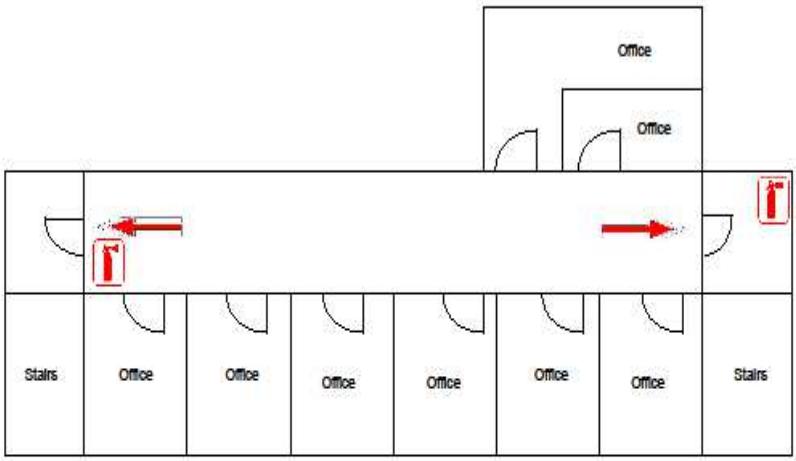
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists EPA Method 8315 but client wanted EPA Method 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. Laboratory Floor Plan





Legend

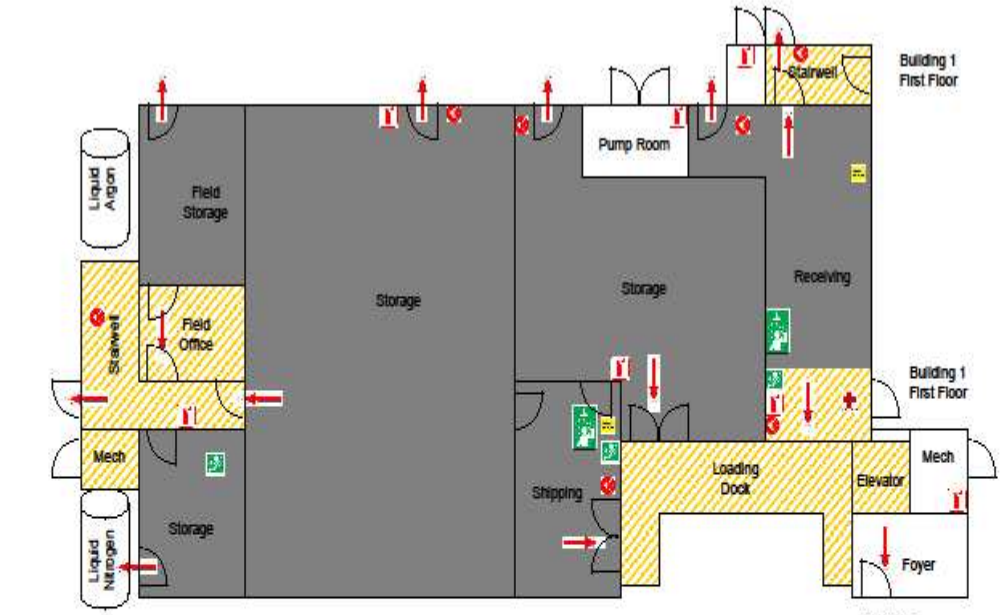
Fire Alarm
Fire Extinguisher
Fire Blanket
Emergency Shower
Eye Wash
First Aid Kit
Exits
Spill Kit

Savannah Division – Building 1 Second Floor
2,200 Square Feet

Legend

Non-Laboratory Area
Dual Use Area
Laboratory Area

FSA003:12.10.14:0



Savannah Division – Building 2 First Floor
8,400 Square Feet

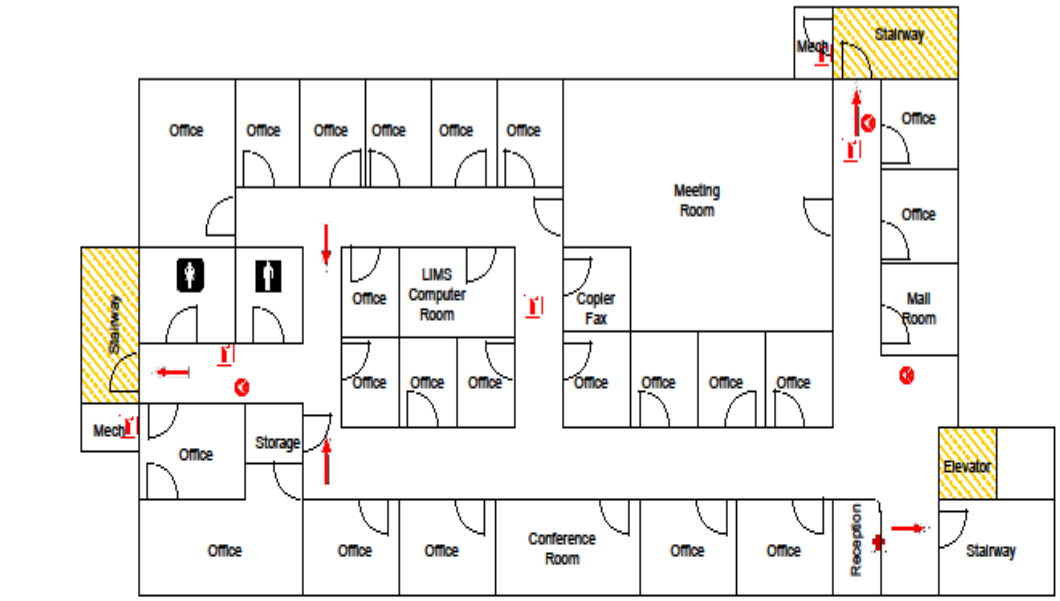
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA004:12.10.14:0



Legend

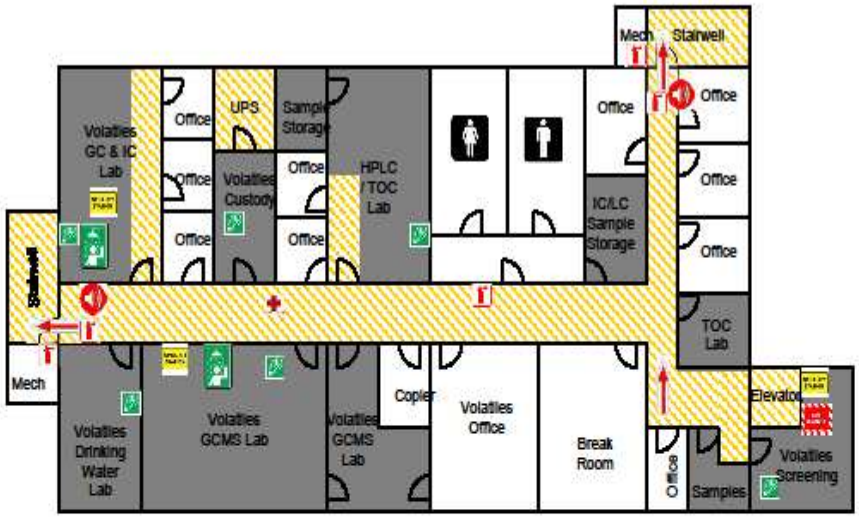
- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Savannah Division – Building 2 Second Floor
8,400 Square Feet

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA005:12.10.14:0



Savannah Division – Building 2 Third Floor
8,400 Square Feet

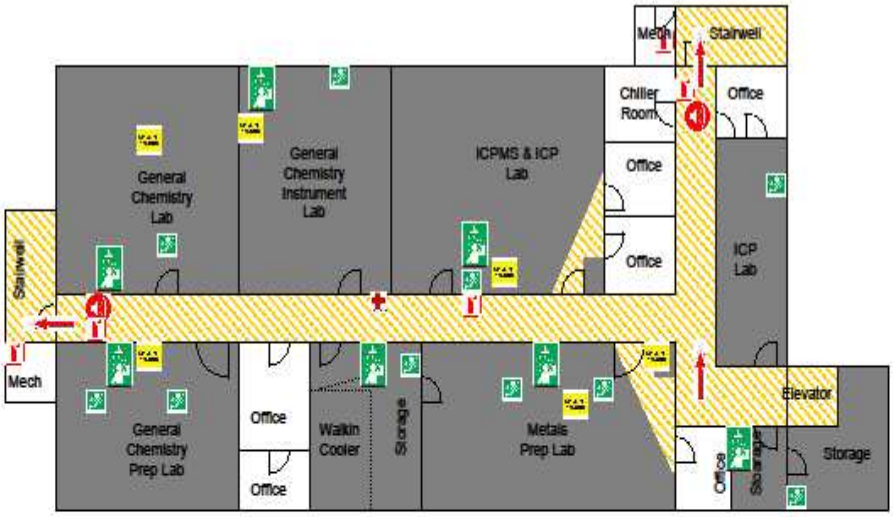
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA006:11.23.15:1



Savannah Division – Building 2 Fourth Floor
8,400 Square Feet

Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA007:12.10.14:0

Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

Glossary:

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:
The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (TNI)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (TNI)

Accuracy:
The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Aliquot:
A representative portion of the sample, standard, or reagent.

Analyst:
The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

Analyte:
The element, molecule, or compound that is being measured in a given procedure. Also referred to as a parameter.

Analytical Method:
Defines the sample preparation and instrumentation procedures that must be performed to determine the quantity of analyte in a sample.

Analytical Sequence:
The order in which calibration standards, verification standards, QC items, and samples are analyzed.

Analytical Spike:
Addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anion:
A negatively charged ion.

Anomaly:
Anomalous situations that are out of the ordinary but are not necessarily a method deviation and are not definitive enough to require a CAR are documented in the Non-Conformance Module. The use of the grand mean exception would require initiation of an Anomaly NCM.

Aromatic:
Relating to the six-carbon-ring configuration of benzene and its derivatives.

Assessment:
The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Assessment Team:
The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (TNI)

Assessor:
One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Background Correction:
A technique to compensate for variable background contribution to the instrument signal and the determination of trace metals.

Batch:
Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias:
The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank:
A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Blind Sample:
A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (TNI)

Calibration Check Compounds (CCC):
Term used in conjunction with SW-846, Method 8260 and 8270 to refer to the compounds in which the percent RSD is evaluated against method-prescribed criteria to decide the validity of a calibration.

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Method:
A defined technical procedure for performing a calibration. (TNI)

Calibration Standard:
A substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain of Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (TNI)

Cation:
A positively charged ion.

Chemical Analysis:
Any of a variety of laboratory methods used to evaluate the concentrations of compounds and elements present in an environmental sample.

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them.

Client Complaint:
A complaint is a situation where dissatisfaction is expressed with the service provided by the laboratory.

Composite Sample:
Portions of material collected from more than one spatial location or at different times that are blended and submitted for chemical analyses. Composite samples can provide data representative of a large area with relatively few samples. However, the resulting data are less accurate with regard to the concentrations of contaminants detected in a specific location, because they represent average values.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites.

Compromised Samples:
Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (TNI)

Concentration:
The mass of analyte per unit mass or volume of sample. Common units of concentration for environmental analyses are microgram per liter or kilogram (ug/L or ug/kg) and milligrams per liter or kilogram (mg/L or mg/kg).

Confidence interval:
For normally distributed (random) data, the intervals where 68%, 95%, and 99% of the data fall. 68% of the data should fall within 1 standard deviation of the mean, 95% of the data should fall within 2 standard deviations of the mean, and 99% of the data should fall within 3 standard deviations of the mean.

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures
(TNI)

Conformance:
An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Continuing Calibration Verification (CCV) Standard:
A mid-concentration analytical standard run periodically to verify the calibration of the analytical instrument. Also known as continuing calibration check (CCC).

Contract Laboratory Program (CLP):
A nationwide laboratory network established by the USEPA, structured to provide legally defensible analytical results to support USEPA enforcement actions or other requirements of the use community. The CLP incorporates a level of quality assurance appropriately designed for the intended usage of the data.

Control Limits:
Accuracy or precision ranges that determine whether the experimentally determined results are in control. If the results are within the acceptance ranges, the results are said to be in control; if the results are outside the limits, they are said to be out-of–control.

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Corrective Action Report (CAR):
The CAR form is used in situations where a recurring problem or breakdown in systems is observed and warrants a more thorough investigation than a single-event NCR. CARs may be initiated from: a specific nonconformance situation (NCM), an observed trend or frequency of events that warrant corrective action, an audit finding, etc.

Correlation Coefficient:
A number (r), which indicates the degree of dependence between two variables (concentration and response). The more dependent the variables are, the closer the value is to one. This value is used to evaluate the straightness of a line, (the linearity of the instrument).

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (TNI)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Data Validation:
An evaluation of laboratory data quality based on a review of the data deliverables. This process involves procedures verifying instrument calibration, calibration verification, and other method-specific performance criterion.

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability (DOC):
Procedure to establish the ability to generate acceptable accuracy and precision. This is done initially upon starting a new method and then continues each year the method is performed.

Detection Limit:
The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (TNI)

Direct Aqueous Injection (DAI):
A technique in which an aliquot of the aqueous sample or aqueous leachate is injected directly into the gas chromatograph with no prior sample preparation.

Disposal:
Final placement or destruction of wastes. Disposal may be accomplished through the use of landfills, treatment processes, etc.

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:
The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

E. coli:
Bacteria giving a positive total coliform response and possessing the enzyme B-glucuronidase, which cleaves the fluorogenic substrate MUG, resulting in the release of a fluorescent product when viewed under long-wavelength UV light.

Environmental Detection Limit (EDL):
The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (TNI Radioanalysis Subcommittee)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Extractable Organics:
Semivolatiles (base/neutral and acid extractable compounds) and pesticide/polychlorinated biphenyl compounds that can be partitioned into an organic solvent from the sample matrix and are amenable to gas chromatography (GC).

Fecal Coliforms:
A subset of total coliforms that grow and ferment lactose at an elevated incubation temperature (44.5℃) and are also referred to as thermotolerant coliforms. Fecal coliforms produce colonies that appear in various shades of blue, domes and glistening, ranging in size from pinpoints to several millimeters. This group consists of mostly E. Coli (EC) but also includes some other enterics. Fecal coliforms are a more specific indicator organism for contamination. This type of bacteria is associated with the fecal material of warm-blooded animals.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (TNI)

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field Control Samples:
General term assigned to field-generated replicates (duplicates/splits/spikes), blanks, background/upgradient samples, etc.

Field Duplicate Sample:
Independent sample collected at approximately the same time and place, using the same methods as another sample. The duplicate and original sample are containerized, handled, and analyzed in an identical manner.

Field of Testing:
TNI's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (TNI)

Filtrate:
A filtered liquid.

Filtration:
The physical removal of solid particles from a liquid wastestream by passing the liquid across a filter medium, which serves as a barrier to the solid material.

Finding:
An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (TNI)

Gas Chromatography/Mass Spectroscopy (GC/MS):
Two distinct analytical techniques used to separate and identify organic compounds: the GC is used for the separating portion and the MS is used as the detection portion of an analysis. Both techniques are typically performed by a single instrument.

Good Laboratory Practices (GLP):
Formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729.

Heavy Metals:
In reference to environmental sampling, typically identified as the following trace inorganics: cadmium, lead, mercury, silver, etc. (all metals of health concern). Heavy metals can cause biological damage if consumed at low concentrations and tend to accumulate in the food chain.

Heterotrophic Bacteria:
A large group of bacteria that obtain energy by oxidizing organic matter. Coliform bacteria are a subset of this group.

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Homogeneous:
The quality of uniform composition.

Initial Calibration Verification (ICV):
A mid-concentration analytical standard run immediately after the calibration to verify the calibration of the analytical instrument. Also known as initial calibration check (ICC).

Inorganic Chemicals:
Chemical substances of mineral origin, not of basically carbon structure.

Inquiry:
A question or request for information about the service provided by the laboratory.

Inspection:
An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Instrument Blank:
A blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Instrument Detection Limit (IDL):
The minimum amount of a substance that can be measured on a specific instrument, with a specified degree of confidence that the amount is greater than zero. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. An IDL value, by definition, has an uncertainty of ±100%. The IDL thus represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Instrument Performance Check Solution (IPC):
A solution of one or more method analytes, surrogates, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

Intermediate or Secondary Stock Standard:
A solution made from two or more stock standards. A secondary standard may also be a certified solution purchased from a vendor as a mixture of several target analytes. Also known as a source reagent in TALS if purchased and an intermediate reagent if prepared in the lab.

Internal Standard:
A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration:
Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:
A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Response:
Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

Job number:
A sequential number that is assigned to each client's samples upon receipt into the laboratory. This log number provides the primary means of associating the samples to the client.

Laboratory:
A defined facility performing environmental analyses in a controlled and scientific manner. (TNI)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (TNI)

Laboratory Duplicate:
Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (TNI)

Laboratory Fortified Blank (LFB):
An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements at the required method detection limit. The percent recovery (accuracy) result for the LFB must fall within the limits listed in the TALS. Also referred to as a laboratory control standard (LCS).

Laboratory Fortified Sample Matrix (LFM):
An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations. The percent recovery (accuracy) result for the LFM must fall within the limits listed in the TALS. Also referred to as a matrix spike (MS).

Laboratory Fortified Sample Matrix Duplicate (LFMD):
A replicate laboratory fortified sample matrix.

Laboratory Performance Check Solution (LPC):
A solution of selected method analytes used to evaluate the performance of the instrumental system with respect to a defined set of method criteria.

Laboratory Quality Manual (LQM):
A document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system. Also referred to as the Quality Assurance Manual (QAM) or Quality Assurance Plan (QAP).

Laboratory Reagent Blank (LRB):
An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Also referred to as a method blank (MB).

Leachate:
The liquid portion of a sample that passes through a 0.6µm filter in the initial evaluation of the percent solids, or the liquid that passes through a 0.6µm filter after the sample has been subjected to the TCLP. The liquid produced by subjecting the sample to the SPLP method.

Least Squares Regression (1^{st} Order Curve):
The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):
An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Liquid phase:
The portion of the sample that passes through the 0.6-0.8□m filter when subjected to a pressure of 50psi during the TCLP or SPLP process.

Manager (however named):
The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (TNI)

Mass Spectrometry (MS):
A detection instrument that differentiates compounds by their differences in mass, or mass fragments. The basic components of the MS are the ion source and lenses, the mass filter (quadrapoles), and the electron multiplier. The ion source and lenses create the ions and propel them on a consistent path to the quadrapoles. The quadrapoles filter the ions that are produced in the source, allowing them to continue to the electron multiplier, where the ions are collected and the signal sent to the data system.

Mass Spectra:
A graphical representation of the abundance of the mass ions produced when a compound is detected by mass spectrometry. The mass spectrum is essentially a fingerprint of the compound and along with the retention time of the compound provides excellent qualitative information about the presence of the compound.

Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Duplicate (MD):
Duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (spiked sample or fortified sample):
Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:
A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (TNI)

Method Detection Limit:
The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)
The MDL is defined as:

$$MDL = SD \otimes t(0.99)$$

SD = standard deviation of the replicates
t(0.99) = Student's t-Value at the 99% confidence level for number of replicates

Most Probable Number (MPN):
An estimate of the mean density of coliforms in a sample based on certain probability formulas.

National Environmental Laboratory Accreditation Conference (NELAC):
A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (TNI)

National Environmental Laboratory Accreditation Program (NELAP):
The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (TNI)

Neat standard:
A pure compound, element, or salt that contains the target analyte. The purity, usually expressed as a percent, of the neat standard must be known. Also known as a source reagent in TALS.

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (TNI)

NELAC Standards:
The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (TNI)

Non-conformance:
Any occurrence that prevents the lab from delivering data that is compliant with the control criteria published (or incorporated by reference) in an applicable QA plan. The Non-conformance Module is used to document nonconformance conditions and to specify the necessary action(s) taken to correct the specific problem.

Organic:
Referring to or derived from living organisms; any compound containing carbon.

Parts Per Billion (ppb):
One part of analyte per billion parts of sample. For aqueous samples, a ppb is equivalent to ug/L; for soils, ug/kg.

Parts Per Million (ppm):
One part of analyte per million parts of sample. For aqueous samples, a ppm is equivalent to mg/L; for soils, mg/kg.

Peak Gaussian Factor (PGF):
A means to measure peak symmetry and monitoring retention time drift over time. Critically evaluate peak in the instrument performance check sample, and calculate the PGF as follows,

$$PGF = \frac{1.83 \otimes W(1/2)}{W(1/10)}$$

where:
W(1/2) is the peak width at half height
W(1/10) is the peak width at tenth height

Percent Recovery:
Percent recovery is used to assess accuracy and is calculated:

$$\%REC = \frac{C\,\text{exp}\,erimental}{Cknown} \otimes 100$$

where:
$C_{experimental}$ = experimentally determined concentration
C_{known} = known or theoretical concentration

Percent Solids:
The proportion of solid in a soil sample determined by drying an aliquot of the sample.

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (TNI)

Performance Based Measurement System (PBMS):
A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (TNI)

pH:
A numerical designation of relative acidity or basicity (Alkalinity). A pH of 7 indicates neutrality; lower values indicate increasing acidity; high values indicate increasing alkalinity.

Precision:
The agreement between two or more experimentally determined results. Precision is routinely expressed as the relative percent difference between two results. Precision is not routinely used as a measurement to determine if the analysis is in control but may be required for certain programs and agencies.

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (TNI)

Post-Digestion Spike:
Addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed.

Preservation:
Refrigeration and or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample. Methods used to retard degradation of chemical analytes within samples by inhibiting decomposition by biological action, chemical reactions, and reducing sorption effects. Methods include limiting headspace, chemical, acid, or base addition, protection from light, cooling, etc.

Precision:
The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation:
Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (TNI)

Preventive Action:
The pro-active process of noting and correcting a potential problem before it happens due to a weakness in a system, method, or procedure.

Procedural Standard Calibration:
A calibration method where aqueous calibration standards are prepared and processed (e.g., purged, extracted, and/or derivatized) in exactly the same manner as a sample. All steps in the process from addition of sampling preservatives through instrumental analyses are included in the calibration. Using procedural standard calibration compensates for any inefficiency in the processing procedure.

Proficiency Testing:
A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Quality Assurance:
An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual:
A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System:
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

Quantitation Limit (QL):
The lowest point at which a substance can be quantitatively measured with a specified degree of confidence using a specific method. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL) or Reporting Limit (RL).

Quantitation Limits:
The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (TNI)

Range:
The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data:
Any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports

specifying inclusion of “raw data” do not need all of the above included, but sufficient information to create the reported data.

Reagent:
A material that is used in a process or analysis but is not directly related to the measured analyte concentration.

Reagent Blank (method reagent blank):
A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:
A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (TNI)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Regulatory Threshold Limit:
The concentration of analyte in the TCLP leachate at which the sample is deemed hazardous.

Relative Percent Difference:
The relative percent difference is calculated between the concentrations of two spikes or sample duplicates:

$$\%RPD = \left| \frac{(C1 - C2)}{\frac{C1 + C2}{2}} \right| \otimes 100$$

Where:
C_1 = concentration of the sample or spike
C_2 = concentration of the sample duplicate or spike duplicate

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (TNI)

Reporting Limit (RL):
Defines the lowest concentration that can be reported with reasonable certainty that the result falls within the laboratories’ accuracy and precision limits. Also referred to as the practical quantitation limit or PQL, the RL is usually defined as the lowest point in the calibration curve or the sample equivalent concentration of the lowest point in the calibration curve.

Representativeness:
A qualitative measure of the extent to which a sample(s) acquired from a medium describes the chemical characteristics of that medium.

Requirement:
Denotes a mandatory specification; often designated by the term “shall”. (TNI)

Resolution:
Also known as separation, or percent resolution. The separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smallest peak being resolved, and multiplied by 100.

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the “cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal. (TNI)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (TNI)

Sample:
A portion of material collected for chemical analyses. Note that a sample is identified by a unique sample number and that the term and the number may apply to multiple sample containers, if a single sample is submitted for a variety of chemical analyses.

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Sampling and Analysis Plan (SAP):
A formal document describing the detailed sampling and analysis procedures for a specific project.

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.990.

Secondary or Intermediate Stock Standard:
A solution made from two or more stock standards. A secondary standard may also be a certified solution purchased from a vendor as a mixture of several target analytes. Also known as a source reagent in TALS if purchased and an intermediate reagent if prepared in the lab.

Selectivity:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Semivolatile Organics:
Compounds that are amenable to analysis by extraction of the sample with an organic solvent. The term semivolatile organic is used synonymously with base/neutral/acid (BNA) compounds.

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Solvent:
The organic liquid used to extract the compounds of interest out of the sample matrix. The solvent is also used to dissolve (put into solution) standards. In general, the solvent used to prepare the standards is also used to extract the samples. A good rule of thumb is that “like dissolves like”, that is, a solvent must be similar in chemical structure to the compound that is being extracted or being dissolved. For most organic extractions, the solvent should also not be miscible (dissolves in all proportions) with water.

Spike:
A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period. (TNI)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of TNI and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):
A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Stock standard:
A solution made from one or more neat standards. The stock standard will usually have a high concentration, usually higher than 1000mg/L (1000ug/mL). This standard can also be purchased from a certified vendor. Also known as a source reagent in TALS.

Storage Blank:
A blank matrix stored with field samples of a similar matrix.

Supervisor (however named):
The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (TNI)

Surrogate:
A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Suspended Metals:
The concentration of metals determined in the portion of a sample that is retained on a 0.45-µm filter. (The concentration of suspended metals may also be calculated from the difference between the total metals sample results minus the dissolved metals sample results.)

Systems Audit (also Technical Systems Audit):
A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

System Performance Check Compounds (SPCCs):
Term used in conjunction with SW-846, Method 8260 and 8270, to refer to the compounds in which the response factor (RF) is evaluated against method-prescribed criteria to decide the validity of a calibration.

Target Analyte List (TAL):
Refers to the Contract Lab Program (CLP) list of inorganic analytes that includes metals and cyanide. May also refer to any general list of inorganic target analytes.

Target Compound List (TCL):
Refers to the Contract Lab Program (CLP) list of organic compounds that includes volatiles (GC/MS), semivolatiles (GC/MS), and pesticides and PCBs (GC/EC). May also refer to any general list of organic target compounds.

Technical Director:
Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (TNI)

Test:
A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:
An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (TNI)

Total Coliforms:
Gram-negative, facultative anaerobic rod-shaped enteric bacteria that ferment lactose to produce colonies with a metallic sheen (yellow to green) when viewed under a fluorescent lamp or acid and gas within 48 hours incubated at 35℃. All bacteria possessing the enzyme B-D-galactosidase, which cleaves the chromogenic substrate ONPG, resulting in release of a chromogen that produces a color change in the sample. They are used as an indicator of contamination in samples although some total coliform bacteria are found naturally in environmental samples. This type of bacteria is commonly found in the intestines of humans.

Total Metals:
Concentration of metals determined in an unfiltered water sample which is preserved (acidified) in the field, transported to the laboratory, and then follows a rigorous digestion.

Total Recoverable Metals:
Concentration of metals in an unfiltered water sample which is preserved (acidified) in the field and transported to the lab, which then performs the digestion with hot dilute mineral acid. This preparation method is typically utilized for drinking water samples and TCLP extracts.

Toxic Substances Control Act (TSCA):
The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (TNI)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Trip Blank:
Samples prepared by adding clean, analyte-free water to sample containers for analysis for volatile organics. Preservatives are added to the blank, and the containers are sealed prior to the sampling trip. Trip blanks are transported with empty sample containers to the site of work and remain sealed until analyzed with collected environmental samples. Trip blanks permit evaluation of contamination generated from sample containers or occurring during the shipping and laboratory storage process.

Tune:
To adjust the parameters of the mass spectrometer in order to meet the mass calibration criteria.

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

United States Environmental Protection Agency (EPA):
The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:
The process of substantiating specified performance criteria. (EPA-QAD)

Verification:
Confirmation by examination and provision of evidence that specified requirements have been met. (TNI)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Volatile Organic Compound (VOC):
An organic compound that is amenable to purge and trap analysis. In general, VOC have low boiling pints (<200^{o}C), high vapor pressures (tend to evaporate easily as low temperatures), and have low molecular weight (generally less than 300amu).

Work Cell:
A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Working Standard:
The standard that is analyzed on the instrument or using the analytical procedure. Also known as an intermediate reagent in TALS.

Acronyms:

ACRONYM	DEFINTION
A2LA	American Association for Laboratory Accreditation
AA	Atomic Absorption
AFCEE	Air Force Center for Environmental Excellence
AL	Action Level
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
bgs	Below Ground Surface
BNA	Base, Neutral, Acids (Semivolatile Organics)
BOD	Biochemical Oxygen Demand
BS	Blank Spike
BSD	Blank Spike Duplicate
BTEX	Benzene, Toluene, Ethylbenzene, Xylenes
BTU	British Thermal Unit
CA	Corrective Action
CAA	Clean Air Act
CAR	Corrective Action Report
CBOD	Carbonaceous Biochemical Oxygen Demand
CCB	Continuing Calibration Blank
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CDC	Continuing Demonstration of Capability
CDOC	Continuing Demonstration of Capability
CDQO	Chemical Data Quality Objective
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act

ACRONYM	DEFINTION
MRF	Method Request Form
MRL	Method Reporting Limit
MS	Mass Spectrometer
MS	Matrix Spike
MS/MS	Tandem Mass Spectrometry
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
MW	Monitoring Well
NBS	National Bureau of Standards
NCASI	National Counsel for Air and Stream Improvement, Inc.
NCM	Non-Conformance Module
NCR	Non-Conformance Report
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
nm	Nanometer
NPD	Nitrogen – Phosphorus Detector
NPDES	National Pollutant Discharge Elimination System
NPW	Non-Potable Water
ORO	Oil Range Organics
OSHA	Occupational Safety and Health Administration
OSTR	Outstanding SOP Training Report

ACRONYM	DEFINTION
CF	Calibration Factor
CFR	Code of Federal Regulations
CLLE	Continuous Liquid-Liquid Extraction
CLP	Contract Laboratory Program
COA	Certificate of Analysis
COC	Chain of Custody
COD	Chemical Oxygen Demand
CRDL	Contract Required Detection Limit
CRF	Change Request Form
CRQL	Contract Required Quantitation Limit
CSM	Corporate Safety Manual
CU	Custody
CVAA	Cold Vapor Atomic Absorption
CWA	Clean Water Act
DAI	Direct Aqueous Injection
DFTPP	Decafluorotriphenylphosphate
DM	Department Manager
DO	Dissolved Oxygen
DOC	Demonstration of Capability
DOD	Department of Defense
DOD QSM	Department of Defense Quality Systems Manual
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data Quality Objective
DRO	Diesel Range Organics
DU	Duplicate
DUP	Duplicate
DW	Drinking Water
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
EDQM	Environmental Data Quality Management
EHS	Environmental Health and Safety
EHSM	Environmental Health and Safety Manual

ACRONYM	DEFINTION
PAH	Polynuclear Aromatic Hydrocarbon
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
PCB	Polychlorinated Biphenyl
PDA	Photodiode Array
PDS	Post Digestion Spike
PE	Performance Evaluation
PGF	Peak Gaussian Factor
PID	Photoionization Detector
PM	Project Manager
PNA	Polynuclear Aromatic Hydrocarbon
PP	Project Plan
ppb	Parts Per Billion
PPE	Personnel Protective Equipment
PPL	Priority Pollutant List
ppm	Parts Per Million
ppq	Part Per Quadrillion
ppt	Parts Per Trillion
PQL	Practical Quantitation Limit
PRG	Preliminary Remediation Goals
PT	Proficiency Test
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl Chloride
PW	Potable Water
PWS	Public Water System
QA	Quality Assurance
QAM	Quality Assurance Manager
QAM	Quality Assurance Manual
QAMP	Quality Assurance Management Plan
QAN	Quality Assurance Navigator
QAP	Quality Assurance Plan
QAPjP	Quality Assurance Project Specific Plan
QAPP	Quality Assurance Project Plan
QAS	Quality Assurance Specialist

ACRONYM	DEFINTION
ELCD	Electrolytic Conductivity Detector
EPA	U.S. Environmental Protection Agency
ERPIMS	Environmental Resources Program Information Management System
eV	Electron Volt
FID	Flame Ionization Detector
FPD	Flame Photometric Detector
GALP	Good Automated Laboratory Practices
GC	Gas Chromatograph or Gas Chromatography
GC/MS	Gas Chromatograph/Mass Spectrometer
GE	General
GFAA	Graphite Furnace Atomic Absorption
GLP	Good Laboratory Practices
GPC	Gel Permeation Column (Gel Permeation Chromatography)
GRO	Gasoline Range Organics
HAA	Haloacetic Acids
HAPS	Hazardous Air Pollutants
HAZMAT	Hazardous Materials
HDPE	High Density Polyethylene
HECD	Electrolytic Conductivity Detector
HPLC	High Performance Liquid Chromatography
HRGC/HRMS	High Resolution Gas Chromatography/Hugh Resolution Mass Spectrometry
HT	Holding Time
HTRW	Hazardous, Toxic, and Radioactive Waste
HTV	Holding Time Violation
IC	Ion Chromatography
IC/EC	Ion Chromatography/Electric Conductivity
IC/MS	Ion Chromatography/Mass Spectrometer

ACRONYM	DEFINTION
QC	Quality Control
QCS	Quality Control Sample
QCSR	Quality Assurance Summary Report
QL	Quantitation Limit
QMP	Quality Management Plan
QSM	Quality Systems Manual
RCRA	Resource Conservation Recovery Act
RF	Response Factor
RI	Remedial Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
RRF	Relative Response Factor
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RT	Retention Time
RTW	Retention Time Window
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SD	Standard Deviation
SD	Sample Dilution
SD	Sample Duplicate
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SG	Semi Volatile Gas Chromatography
SIM	Selected Ion Monitoring
SM	Semi Volatile Mass Chromatography
SOC	Synthetic Organic Compound

ACRONYM	DEFINTION
ICAP	Inductively Coupled Argon Plasma Emission Spectroscopy
ICB	Initial Calibration Blank
ICCS	Interference Calibration Check Sample
ICOC	Internal Chain of Custody
ICP	Inductively Coupled Plasma
ICP/MS	ICP/Mass Spectrometer
ICS	Interference Check Sample
ICV	Initial Calibration Verification
IDC	Initial Demonstration of Capability
IDL	Instrument Detection Limit
IDOC	Initial Demonstration of Capability
IH	Industrial Hygiene
IPC	Instrument Performance Check Standard
IR	Infrared Radiation
IS	Internal Standard
ISO	International Standards Organization
ISTD	Internal Standard
LC	Liquid Chromatography
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LFB	Laboratory Fortified Blank
LFM	Laboratory Fortified Matrix
LFMD	Laboratory Fortified Matrix Duplicate
LIMS	Laboratory Information Management System
LM	Laboratory Manager
LOD	Limit of Detection
LOQ	Limit of Quantitation
LPC	Laboratory Performance Check
LQM	Laboratory Quality Manual
LRB	Laboratory Reagent Blank
LUFT	Leaking Underground Fuel Tank
LUST	Leaking Underground Storage Tank

ACRONYM	DEFINTION
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compound
SPE	Solid Phase Extraction
SPLP	Synthetic Precipitation Leaching Procedure
SR	Shipping and Receiving
SRM	Standard Reference Material
SS	Suspended Solids
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SVOC	Semi Volatile Organic Compound
SW-846	Solid Waste Analytical Protocols
TAL	Target Analyte List
TALS	TestAmerica LIMS System
TAT	Turn-Around-Time
TCL	Target Compound List
TCLP	Toxicity Characteristic Leachate Procedure
TDS	Total Dissolved Solids
TEPH	Total Extractable Petroleum Hydrocarbons
THM	Trihalomethanes
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TM	Technical Manager
TOC	Total Organic Carbon
TOX	Total Organic Halides
TPH	Total Petroleum Hydrocarbons
TRPH	Total Recoverable Petroleum Hydrocarbons
TS	Total Solids
TSD	Thermionic Specific Detector
TSS	Total Suspended Solids
TVPH	Total Volatile Petroleum Hydrocarbons
TVS	Total Volatile Solids
UCL	Upper Confidence Level

ACRONYM	DEFINTION
MB	Method Blank
MB	Microbiology
MBAS	Methylene Blue Active Substances
MCL	Maximum Contaminant Level
MCT	Maximum Conductivity Threshold
MD	Matrix Duplicate
MDL	Method Detection Limit
ME	Metals
µg/L	Microgram per Liter
mg/L	Milligram per Liter
MLG	Method Limit Group
µm	Micrometer
MPN	Most Probable Number

ACRONYM	DEFINTION
UCMR	Unregulated Contaminant Monitoring Rule
US EPA	United States Environmental Protection Agency
USACE	United States Army Corps of Engineers
USDA	United States Department of Agriculture
USGS	United States Geological Service
UST	Underground Storage Tank
UV	Ultraviolet
VG	Volatile Gas Chromatography
VM	Volatile Mass Chromatography
VOA	Volatile Organic Analysis / Volatile Organic Analyte
VOC	Volatile Organic Compound
ZHE	Zero Headspace Extraction

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Savannah performs work from clients located throughout the United States, as well as in some foreign countries. Most states and/or federal agencies maintain a laboratory accreditation program that requires a laboratory to obtain certification with their agency. To obtain certification, a laboratory must maintain an effective quality system that meets the requirements of the agency. Common components of the quality system requirements include maintaining up-to-date standard operating procedures (SOPs) and a Quality Assurance Manual (QAM); participating in a Proficiency Testing (PT) program; performing method detection limit (MDL) studies, initial and continuing demonstrations of capability studies (IDOCs/CDOCs), and internal assessments; and completing an annual renewal application. In addition to the requirements needed for certification, many agencies have specific analytical and/or reporting requirements that laboratories must follow.

Many agencies offer certification via reciprocity. Reciprocity is the acknowledgement of another state and/or agency's certification program. The most common types of reciprocity are homestate reciprocity and TNI (NELAC) reciprocity.

Lab Management, Project Management, Sales & Marketing, and the QA Manager may initiate requests for certification or accreditation. The QA staff completes the administrative tasks associated with the application and maintains the related documents in accordance with SOP SA-QA-001: *Document Control Program.*

Laboratory management has the responsibility and authority to ensure that laboratory operations are in compliance with program and regulatory requirements of the jurisdiction for which laboratory certification/accreditation is sought and maintained.

To perform compliance work in a particular state, the laboratory must maintain certification for the reported analytes. Most accrediting authorities will certify laboratories on a matrix/method/analyte level. For example:

Soil / EPA 8260B / benzene
Water / EPA 624 / toluene

Generally, laboratories must apply and submit supporting documentation (SOPs, MDLs, IDOCs, PTs, etc.) for each individual matrix/method/analyte combination.

1.0 Obtaining Certification

1.1 Certification Application Process

Lab Management, Project Management, Sales and Marketing, and the QA Manager may initiate requests for certification or accreditation. The application is obtained, reviewed, and completed by the QA Manager or designee. Sections of the application may be

distributed as appropriate to various staff members to assist in completion.

The certifying agency's regulations should be carefully reviewed at the time of application to ensure any non-routine requirements are communicated to the laboratory.

The QA Manager consults with Lab Management, Project Management, and the Sales and Marketing Staff to determine if additional methods should be added to the current laboratory certification in order to support existing and future work as the laboratory's capabilities change.

1.2 Reciprocity

Reciprocity is a means of acknowledging another agency's certification via mutual agreements between certifying agencies. Many certifying agencies offer some type of reciprocal certification. The most common types of reciprocity are based on either TNI (NELAC) certification or homestate certification.

Homestate reciprocity refers to another state's certifying agency allowing a laboratory to perform work in that state, provided that the laboratory maintains accreditation within the state in which it resides.

TNI refers to The NELAP Institute which governs the NELAP Standard document outlining Quality System and laboratory functions and requirements. NELAP refers to the National Environmental Laboratory Accreditation Program. Many states will acknowledge a laboratory's TNI accreditation from another state.

Note: Reciprocal agreements between states do not afford a "blanket" certification. To obtain reciprocal certification, a laboratory must still apply for accreditation, submit all required application materials, and receive notification of certification – usually in the form of a certificate - from the reciprocal agency.

1.3 Records Maintenance

A copy of the original application, certificate, and related materials are maintained in accordance with SA-QA-001: *Document Control Program.* Copies of current certifications are kept in the Certifications folder on the Public_QA drive, which is accessible to all laboratory staff. In addition, copies of current laboratory certifications from Savannah and other TestAmerica facilities are maintained in the TotalAccess marketing tool. These documents may be required to support subcontracting and marketing activities.

1.4 Maintaining Certification

Most states require continued evidence of an effective Quality System in order for a laboratory to maintain certification. In addition to annual renewal applications, laboratories are often required to complete bi-annual PT studies with acceptable results obtained for each certifiable matrix/method/analyte combination. Annual MDL and continued demonstrations of analyst capability are also routinely required, in addition to on-site assessments.

1.5 Certification Tools and Records

There are several tools in place to aid laboratory staff in determining what certifications the laboratory maintains and understanding any state-specific analytical and/or reporting requirements.

1.5.1 TotalAccess

Total Access is a tool that can aid in determining which certifications the laboratory maintains. This tool is useful in the pre-project planning process.

1.5.2 State and Project Requirement Summaries

Some states and/or projects have specific analytical and/or reporting requirements. A summary of these requirements is kept in the State and Project Requirement Summary on the Public-QA Drive. These requirements must be reviewed by project management and laboratory staff prior to initiating work. The Project Manager must clearly note in the TALS Worksheet Notes and/or Project Plan if the Project Requirement Summary (PRS) is to be followed.

1.6 Information Resources

1.6.1 Agency Information

The QA staff maintains a controlled access database that lists current contact information for the agency that oversees laboratory certification as well as the regulatory programs that are offered for certification by the agency. This information may be provided as a resource to Lab Management, Project Management, Corporate QA, and the Sales and Marketing staff.

1.6.2 Certification Matrix

The QA Department ensures that the certification matrix maintained on TotalAccess is current.

1.6.3 Certification / Accreditation Maintenance Requirements

Laboratory Management is responsible for ensuring that laboratory operations are in compliance with the regulatory and certification program requirements for the jurisdiction in which certification is maintained.

The QA Department is responsible for maintaining up to date applications and program information including program specific regulations and requirements.

Project Management is responsible for verifying certification of analytes and methods requested by the client prior to accepting work and should be familiar with the state-specific requirements of that state.

1.7 Certifications Listing

At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Authority	Certification Number or Laboratory ID Number
A2LA (DoD ELAP)	0399-01
A2LA (ISO/IEC 17025)	399.01
Alabama	41450
Alaska	UST-104
Arkansas	88-0692
California	3217CA
Colorado	N/A
Connecticut	PH-0161
Florida	E87052
Georgia	803
Georgia EPD	N/A
Guam	09-005r
Hawaii	N/A
Illinois	200022
Indiana	N/A
Iowa	353
Kentucky	90084
Kentucky UST	18
Louisiana	30690
Louisiana	LA160019
Maine	GA00006
Maryland	250
Massachusetts	M-GA006
Michigan	9925
Mississippi	N/A

Authority	Certification Number or Laboratory ID Number
Nebraska	TestAmerica-Savannah
New Jersey	GA769
New Mexico	N/A
New York	10842
North Carolina DENR	269
North Carolina PHL	13701
Oklahoma	9984
Pennsylvania	68-00474
Puerto Rico	GA00006
South Carolina	98001
Tennessee	TN02961
Texas	T104704185-08-TX
USDA	SAV 3-04
Virginia	302
Washington	C1794
West Virginia DEP	94
West Virginia DHHR (DW)	9950C
Wisconsin	999819810
Wyoming	8TMS-Q

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications. Copies of these documents can also be found on the laboratory's public server, in TotalAccess, and in the QA offices.

Revision History

Changes from the previous revision include:

- Updated Title Page and approved signatories.
- Updated Table of Contents.
- Updated SOP Listing.
- Updated references/requirements to incorporate SW846 Update V. Section 3
- Updated employee titles to reflect current designations. Section 4
- Included roles/responsibilities for Corporate ECO and Corporate QA employees. Section 4
- Updated laboratory employee responsibilities to be consistent with QAM template. Section 4
- Revised employee designated to serve as Laboratory Director Deputy to reflect QAM. Previously listed as CSM. Section 4.1
- Updated Corporate and laboratory Organization Charts. Section 4
- Updated laboratory's document control procedure to remove reference to QA Navigator. Replaced with reference to TALS File Shares.
- Added requirement that LD is responsible for reviewing and authorizing final acceptance of the quote for the facility. Section 7.1
- Removed requirement for Contracts Director and Proposal Coordinator to maintain copies of quote. Section 7.1
- Included reference to new Corporate Workshare SOP CA-C-S-001. Section 8
- Revised subcontract process to remove statement that client must provide acknowledgement that samples can be sent to another facility. Section 8
- Revised Workshare process to note that copy of the original COC is housed TALS, as opposed to requiring it to be shipped with the samples. Section 8.1
- Revised process for solvent and acid lot testing to denote that the approval information is located on the SharePoint directory on Oasis. Section 9.3
- Expanded responsibilities and process for receiving of materials and supplies. Incorporated requirements for verification of lots of acids and solvents. Section 9.2
- Removed QA Department as responsible party to maintain software verification. This responsibility lies with the IT Department. Section 9.3
- Expanded section on calibration of support equipment. Section 9.4
- Expanded section on and updated corporate SOP references for Data Recalls and Data investigations. Section 11
- Included reference to corporate Root Cause SOP. Section 12
- Added requirement that the QA Metrics Report is reviewed monthly by laboratory management, Corporate QA, and the EC. Section 13.1
- Expanded section on continuous improvement activities. Section 13
- Updated document control/archival process. Added note that QA records are maintained by the QA Department on the Q-Drive and are a part of the routine network backups. Removed requirement to document access to data with an access log. Removed reference regarding maintaining analytical data in hard copy. Removed reference to I-drive. Removed requirement to maintain run logbook per instrument. Section 14
- Removed reference to Target and MintMiner. Replaced with CHROM and AuditMiner. Section 15
- Expanded technical method audit process to incorporate data authenticity and analyst integrity audit such that each analyst is reviewed over the course of a 2 year period. Section 15
- Added requirement to review all SOPs annually. Section 15 and Section 19
- Added requirement to generate a written response for all PT failures. Section 19

- Revised MDL definition to denote as the value at which the analyst is 99% confident that the true value can be distinguished from blanks, as opposed to greater than zero. Section 19
- Expanded process/requirements for Login Review, First Level Review, and Second Level Review. Section 19
- Revised thermometer verification requirements to require bracketing temperature verifications only when thermometer range of use is greater than 10 degrees. Section 20
- Removed requirement to verify IR gun at a freezing temperature as this temperature is not encountered in normal use. Section 20
- Updated Instrument List. Section 20
- Removed reference to EPA Method 1653 as this method is no longer performed by the laboratory. Section 20 and Section 21
- Added requirement to verify mechanical pipettes daily for DOD. Added requirement to verify digital thermometers quarterly. Section 21
- Added requirements for verification of standards received without a Certificate of Analysis. Expanded process for receipt and documentation of reference standards. Section 21
- Removed text defining the first day of holding time as ending at 24 hours after sampling. Section 22
- Defined authorized test report signatories as the PM and PMA. Section 25
- Removed reference to faxing the final report. Section 25
- Added note that when NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Section 25
- Updated Laboratory Floor Plans.
- Updated location of laboratory certificates to reference Public_QA drive and TAACM. Removed reference to Public_G drive and Oasis. Updated Certifications Listing to reflect current certifications. Appendix 3

MICROEXTRACTABLES BY GC/ECD

(Methods: EPA 504.1 and 8011)

Approvals (Signature/Date):

Andrea Teal — May 27, 2015
Andrea Teal — Date
Quality Assurance Manager

Whitney Palefsky — April 23, 2015
Whitney Palefsky — Date
Environmental Health and Safety Coordinator

Kimberly Chamberlain — April 28, 2015
Kimberly Chamberlain — Date
Technical Manager

1.0 Scope and Application

This SOP gives the procedures for the determination of 1,2-Dibromoethane (Ethylene dibromide, EDB), 1,2-Dibromo-3-chloropropane (Dibromochloropropane, DBCP), and 1,2,3-Trichloropropane (1,2,3-TCP) in water samples by microextraction and gas chromatography/electron capture detection (GC/ECD).

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Thirty-five milliliters of sample are extracted with two milliliters of hexane. The extract is analyzed by gas chromatography utilizing dual capillary columns and dual electron capture (EC) detectors. Calibration standards are extracted and analyzed in the same manner as the samples.

This SOP is based on the following methods: EPA 504.1 and EPA 8011.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hexane and methanol must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences. Addition of sodium sulfate may be necessary to break up emulsions should they form during the extraction process.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Dibromochloromethane (DBCM) is a common disinfection byproduct in chlorinated drinking waters, often occurring in high concentrations. DBCM elutes closely to EDB, and, at high concentrations, may mask low concentrations of EDB. Adequate separation of DBCM and EDB must be demonstrated each day samples are analyzed.

4.2.4 High concentrations of non-target compounds may make detection and quantification of EDB, DBCP, and 1,2,3-TCP difficult. The electron capture detector is very sensitive to halogenated compounds and produces a very large response for concentrations as low as 1ppb. The results from the EPA 524.2 or EPA 8260 volatiles analysis can provide information about the compounds causing the interference. Some common volatile compounds that elute near the target compounds are tetrachloroethene, dibromochloromethane, chlorobenzene, bromoform, 1,2-dichlorobenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene. Note that very large concentrations of any halogenated solvent may overwhelm the response of the electron capture detector, making detection of the target compounds impossible at the routine reporting limit.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Hexane is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

Methanol is the primary solvent for standards. Hexane is used to extract the compounds from the samples. To minimize evaporation and the chance for exposure:

- store standards in methanol in glass containers with crimp-top caps and vials with Teflon-lined caps or septa
- store material with minimal headspace
- store materials at -10^{o}C or lower
- work under a hood
- if no hood is available, work in a well ventilated area, work quickly, and minimize the number of times the standard container is opened
- wear proper PPE (Personal Protection Equipment). PPE for this procedure includes a laboratory coat, eye protection, and gloves when handling standards, samples, or reagents.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Hexane	Flammable Irritant	500ppm TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

[1]Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Data System – Chemstation software is used to acquire and store data. CHROM software is used to reduce and output data. This software has the capability of processing stored GC data by recognizing a GC peak within any given retention time window and comparing the response of the peak to a reference standard. The software also allows calculation of response factors or construction of a calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

Gas Chromatograph - Agilent 6890 GC with 7683 autosampler, or equivalent, with dual micro electron capture detectors

Columns:

RTX CLP Pest, 30m x 0.32mm ID x 0.50um (Restek)
RTX CLP2, 30m x 0.32mm ID x 0.25um (Restek)
Guard Column Restek 5m x 0.32mm ID (Restek)

The columns are connected to a single injection port via a glass y-splitter and a 5m guard column. The ends of each column are connected to separate detectors. When properly configured, a single injection is split between the two columns to provide simultaneous detection and confirmation of the target compounds.

6.2 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 4 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Glass Transfer Pipettes	Various	Qualitative	Aliquotting sample extract	None	None
Mechanical Eppendorf-Style Pipettes (w/ Tips)	Various	QUANTITATIVE	Dilution Preparation	Monthly (Daily for DOD v5)	Accuracy = 2% Precision = 1%
Volumetric Flask (Class A)	Various	QUANTITATIVE	Calibration Standards Preparation	None (Class A)	None (Class A)
Gas Tight Autosampler Syringe	Various	QUANTITATIVE	Extract Injection; Standard and Spike Preparation	None, if received w/ COA	None, if received w/ COA
Autosampler Vial	2mL	Qualitative	Sample and Standard Containment	None	None
Sample Vial	12mL	Qualitative	Sample and Standard containment	None	None
VOA Vial	40mL 60mL	Qualitative	Standard and Spike containment	None	None
PTFE Squeeze Bottle	500mL	Qualitative	Dispensing Solvents	None	None
Pump-Style Mechanical Pipette	2mL	QUANTITATIVE	Dispensing Hexane	Monthly (Daily for DOD)	Accuracy = 2% Precision = 1%
Graduated Cylinder (Class A)	Various	QUANTITATIVE	QC Preparation	None (Class A)	None (Class A)
Erlenmeyer Flask	500mL	Qualitative	Extract Storage and Drying	None	None
Receiving Tip	10mL	Qualitative	Secondary Concentration Vessel	None	None

6.3 Lab Supplies

Residual (free) chlorine powder pack – HACH catalogue number 1539357 (for a 5mL sample)

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

40mL VOA vial, with sodium thiosulfate dechlorination agent – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041*: Reagent and Standard Materials Procedures.*

Hexane and methanol must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Purchased Reagents

7.2.1.1 Laboratory Reagent Water – ASTM Type II

Note: If laboratory water is unsuitable for analysis of target compounds, purging the water with nitrogen for 30 minutes or boiling the water and purging with nitrogen may reduce contamination.

7.2.1.2 Hexane – residue grade
TALS Name: Hexane(lot number)_
Storage: Room temperature
Expiration:
Unopened: 5 years or manufacturer's expiration date
Opened: 6 months

7.2.1.3 Methanol – residue grade
TALS Name: EX_MEOH
Storage: Room temperature
Expiration:
Unopened: 5 years or manufacturer's expiration date
Opened: 6 months

7.1.4 Sodium Chloride (NaCl) - ACS reagent grade. Purify by heating at 400°C for four hours in a shallow tray.
TALS Name: NACL
Storage: Room temperature
Expiration:
Unopened: 5 years or manufacturer's expiration date

Opened: 5 years or manufacturer's expiration date; 6 months from baking.

7.1.5 Sodium Sulfate - granular, anhydrous - Purify by heating at 400°C for four hours in a shallow tray.
TALS Name: EX_na2SO4
Storage: Room temperature
Expiration:
Unopened: 5 years or manufacturer's expiration date
Opened: 5 years or manufacturer's expiration date; 6 months from baking.

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and attached to TALS.

The standard recipes are included in Attachment 5. This attachment includes stock standards (vendor and part number) used in this SOP, the preparation steps for the intermediate and working standards, and the instructions for assigning expiration dates to the stocks, intermediate, and working level standards.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Samples are routinely collected in 40mL VOA vials containing 75uL of a 40mg/mL solution of sodium thiosulfate de-chlorination agent. The dechlorination agent should be sufficient to remove residual chlorine from the sample. Samples should be collected without headspace.

Note: 40mL VOA vials with HCl preservative may also be used for EPA 8011. This bottle type is consistent with the bottle type used commonly used for analysis of these particular analytes by EPA 8260.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored at in the refrigerator at 0-6°C (less than 6°C but not frozen) until the time of analysis and analyzed within 24 hours of extraction.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives and/or de-chlorination agents. NCMs must be initiated for samples that are received containing headspace.

8.1.1 Preservation Checks – Residual Chlorine

These checks are performed prior to preparation.

8.1.1.1 Mix the sample by inverting and transfer 6mL to a small medicine cup.

8.1.1.2 Add a residual chlorine powder pillow to the sample in the cup and note the presence of a pink color, which indicates the presence of residual chlorine.

If the sample tests positive for residual chlorine, initiate an NCM noting that residual chlorine was present.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An extraction batch consists of up to 20 environmental samples and the associated QC items extracted together within a 24 hour period.

9.1.1 For EPA 504.1 and EPA 8011, the laboratory's default minimum QC items performed for each extraction batch are: a method blank, laboratory control sample (LCS), a low-level LCS (LLCS) at the method detection limit (MDL), a matrix spike (MS) per 10 samples, and a matrix spike duplicate (MSD).

Note: This procedure incorporates extracted calibration standards; therefore, initial and continuing calibration standards are also prepared within the extraction batch. Refer to Attachment 5 for standard preparation instructions.

9.1.2 The routine container supplied for this method is a 40mL container. 35mL is required for extraction. Due to the nature of this procedure, and the need to maintain zero headspace, reduced sample initial volumes must not be used to achieve the required batch matrix spike frequency (i.e., a separate vial is required for to perform each of the native sample, MS, and MSD analyses). As such, an MS/MSD can only be prepared when additional containers are provided.

9.1.3 If there is insufficient sample volume to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of three 40mL VOA vials for matrix spike/matrix spike duplicate and less than two 40mL VOA vials for the additional matrix spike required for batches having greater than 10 samples.

9.1.4 Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Instrument QC

9.2.1 Column Resolution / RL Check

The purpose of the Column Resolution / RL Check is check is to ensure that EDB can be resolved from a common chlorination by-product, dibromochloromethane (DBCM). DBCM is often present at concentrations that are much higher than EDB is expected to occur, and this check is intended to demonstrate that EDB can be detected at the RL of 0.020ug/L in the presence of DBCM at 1.0ug/L, a 50-fold difference in concentrations.

- Prepare, extract, and analyze the Column Resolution Check standard (Table 5).

- Evaluate the chromatogram, inspecting the resolution between DBCM and EDB on both columns.

- The peaks should be resolved at the baseline (a gap in the baseline from the end of the DBCM peak to the beginning of the EDB peak) on both columns.

- If EDB cannot be detected, prepare a new standard and repeat the preparation and analysis. If the repeat analysis still does not meet the criterion, take steps to increase the resolution between these two compounds which may include decreasing the initial temperature and/or reducing the column flow rate.

Note: Do not proceed with the analysis is this check can be met.

9.2.2 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 5 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted.

The initial calibration standard concentrations currently in use in the laboratory are listed in Attachment 5. Refer to Attachment 5 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

Note: This procedure incorporates extracted standards; therefore, initial and continuing calibration standards are prepared within the extraction batch. Once the ICAL is established, subsequent CCVs must be extracted each day samples are extracted. All ICAL, ICV, and CCV standards must be analyzed within 24 hours of extraction.

9.2.2.1 ICAL Criteria

The preferred method of quantitation is the average response factor. The relative standard deviation (%RSD) of the calibration standards must be <10% for EPA 8011 and <20% for EPA 504.1 for the initial calibration curve to be acceptable.

If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. If the regression curve option is chosen, the regression coefficient (r^2) must be greater than or equal to 0.990 to be acceptable.

If these criteria are not met, then re-calibration is required before sample analysis can proceed.

9.2.3 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified after the initial calibration is established, prior to any sample analyses, in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to Level 5 of the ICAL. Refer to Attachment 5 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

The ICV must be within 30% of the true value to be acceptable.

Note: If the LCS is prepared from a second source standard it can be used to satisfy the ICV criteria.

9.2.4 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The method blank for this method is analyzed and evaluated in lieu of instrument or calibration blanks.

Additional instrument blanks may be analyzed after samples with high levels of target or non-target compounds to mitigate and evaluate the analytical system for carry-over.

9.2.5 Continuing Calibration Verification

The initial calibration curve must be verified at the beginning and end of every 12-hour clock for EPA 504.1 and at the before and after every 20 samples analyzed for EPA 8011.

The concentration of the standard should be varied, such that several points of the calibration range are verified.

The CCV must be within 30%D to be acceptable for EPA 504.1.

The CCV must be within 20%D to be acceptable for EPA 8011.

9.2.6 Surrogate

This procedure uses a surrogate compound to evaluate the extraction process. Pentachloroethane is the surrogate used for this procedure. Another surrogate compound may be used provided it produces consistent results within method-defined criteria.

Prior to preparation, this surrogate is added to all samples and QC items. The concentration of the surrogate is the same in all field samples and QC samples. A concentration of 0.50ug/L is used.

The percent recovery of the surrogate in all field samples and QC samples must be within the limits listed in the Method Limit Groups (MLGs) in TALS. If the percent recovery is

outside of this range, the analysis of the sample must be repeated. Repeated failure of the surrogate percent recovery may indicate re-extraction is necessary.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

Samples, calibration standards, and QC items are subjected to the same extraction and analytical procedures.

10.1.1 Remove the samples from storage and scan them into the SG Department. Allow them to warm to room temperature.

10.1.2 Gather and label one 40mL VOA vial for each calibration standard and QC item. Add 35mL reagent water to each of the labeled VOA vials. Prepare the calibration standards in accordance with Attachment 5. Prepare the QC items in accordance with Section 10.2.

10.1.3 Scan each sample into the prep batch and complete the information for the calibration standards and the QC items for the batch.

10.1.4 Inspect the samples for large air bubbles, the presence of large amounts of sediment, and other anomalies, and, if present, contact the Department Manager to determine the course of action.

In the absence of any additional guidance, use the following:

- If the sample contains air bubbles, notify the Project Manager via a Nonconformance Memo (NCM) and proceed with the analysis.
- If the sample contains large amounts of sediment, pour off the liquid above the sediment into a tared 40mL vial and proceed with the analysis. Notify the Project Manager via an NCM.

10.1.5 Working with each sample in turn, invert the sample vial three times and open the cap. Pipette 6mL of sample from each 40mL sample vial and transfer to a labeled, 25mL plastic cup. Cap the vial. Repeat for the remaining samples.

Add a residual chlorine powder pack to the 6mL of sample that was transferred to the plastic cup. Note whether the residual chlorine test is positive (pink color forms) or negative (no color forms) on the prep sheet. If residual chlorine is present, an NCM must be initiated.

10.1.6 Weigh and record the weight of the capped sample vials (after removing 6mL) to the nearest 0.1 grams on the prep sheet.

10.1.7 Add the surrogate spiking solution to each sample and QC item as follows:

- draw 35uL of the surrogate spiking solution into a 50uL syringe
- inject the surrogate spiking solution, through the vial septum, under the surface of the sample
- invert the sample once to mix
- repeat for the remaining samples and QC items.

10.1.8 Add the 504 Spike Solution to each LCS/LCSD and MS/MSD item as follows:

- draw 35uL of the 504 Spike Solution into a 50uL syringe
- inject the 504 Spike Solution, through the vial septum, under the surface of the sample
- invert the sample once to mix
- repeat for the remaining QC items.

Add the WS#1 Spike Solution to the LLCS as follows:

- draw 10uL of the WS#1 Spike Solution into a 10uL syringe
- inject the WS#1 Spike Solution, through the vial septum, under the surface of the sample
- invert the sample once to mix
- repeat for the remaining QC items.

10.1.9 Working with one vial at a time, quickly remove the cap, add 6g of purified sodium chloride (NaCl), then pipette 2.0mL hexane into the vial for each standard, sample, and QC item. Recap the sample containers and gently swirl until the NaCl has dissolved.

10.1.10 Shake the vials for approximately three minutes by hand, or for approximately 5 minutes using a shaker table set on high.

10.1.11 Allow the hexane and water layers to separate.

10.1.12 Remove the cap and carefully transfer approximately 400uL of the extract (i.e., hexane; top layer) into a GC autosampler vial fitted with a 400uL insert. Cap the vial. The extract is now ready for analysis.

Note: If an emulsion forms in the solvent layer, add small amounts (~0.1g) of purified sodium sulfate to the extract, letting the crystals fall gently through the emulsion/solvent layer.

If the sodium sulfate does not break the emulsion, centrifuge the sample for approximately three minutes. The centrifuge is located in the 508 Prep Laboratory. If centrifugation does not clear up the emulsion, freeze the sample to separate the water layer and solvent layer.

Invert the remaining sample and vials and store in the refrigerator at 0-6^{o}C until analysis is completed. The extracts must be analyzed within 24 hours of extraction.

10.1.13 Determine the volume of sample as follows:

- remove the cap and pour the sample into a separatory funnel to separate the hexane from the water. Discard the water sample (bottom layer) down the sink then pour the remaining hexane (top layer) into the flammable waste container.
- "flick" the sample vial several time to remove the remaining drops of water
- recap the vial, weigh the vial, and record the weight of the empty sample container on the prep sheet to the nearest 0.1g.

10.1.14 Calculate the volume of sample, assuming that 1.0g of sample is equal to 1.0mL of sample.

$$V = (W_1 - W_2) \otimes \frac{1.0mL}{g}$$

Where:
V = volume of sample extracted (mL)
W_1 = weight of vial, cap, and sample (g) (Section 9.6)
W_2 =weight of empty vial and cap (Section 9.13)

Record the volume of sample on the prep sheet.

10.1.15 Complete the TALS prep sheet.

10.2 QC Sample Preparation

Refer to Section 9.1 for the minimum QC items to be prepared with each preparation batch of twenty or fewer field samples. Additionally, the Resolution Check / RL Standard and a mid-level CCV (or multi-point ICAL) are also required.

Note: The LCS/LCSD are prepared using a source different from the source used to the calibration standards and also serve the initial calibration verification standards for an ICAL.

Batch QC samples are processed in the same manner as field samples.

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

Two dissimilar columns are connected to the injection port using a press-tight glass y-splitter and a guard column which provides simultaneous detection and confirmation of the target analytes.

These conditions and parameters are given for guidance and for a starting point if the method is lost in the acquisition computer. The conditions and parameters may be

modified to optimize the analytical system. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the MDL.

GC Parameters
Column1: Restek CLPesticides 30m x 320um, x 0.50um
Column2: Restek CLPesticides 2 30m x 320um, x 0.25um
Injector: 240°C
Mode: Pulsed Splitless
Pressure: 25.14psi
Pulse pressure: 50.0psi
Pulse time: 0.30 minutes
Purge flow: 55.9mL/minute
Purge time: 0.28 minutes
Total flow: 94.9mL/min (hydrogen)

Temperature program
Initial Temp: 55°C
Initial Hold: 1.60 minute
Program Rate: 76.33°C per minute to 95°C, hold 1.00 minutes
60.00°C per minute to 150°C hold 1.29 minutes
Maximum Temp: 320°C

Run Time: Approximately 5.33 minutes

Detector: Dual electron capture
Detector temperature: 305°C
Makeup flow: 80mL/min (nitrogen)

Signal data rate: 10hz

Autoinjector
Sample washes: 0
Sample pumps: 4
Injection volume: 2.0uL
Syringe size: 10uL
PostInj(ection) Solvent A washes: 6
PostInj(ection) Solvent B washes: 6 (use hexane as wash solvent)
Viscosity delay: 0 seconds
Plunger speed: fast
Preinjection dwell: 0.00minutes
Postinjection dwell: 0.00minutes

10.3.1.1 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP SA-QA-008: *Evaluation of Chromatographic Data*. Retention time windows (RTW), i.e., the length of time the instrument will scan for the analyte, must be established initially upon instrument set-up and verified annually.

Retention times (RT), i.e., the elution time of the analyte, are verified daily with the analysis of the ICAL or CCV. The retention time for the CCV must fall within the daily retention time window as defined in SOP SA-QA-008.

10.3.2 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.2. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.3, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5.

10.3.3 Sample Analysis

Remove the extracts from the refrigerator and allow them to come to room temperature.

The sample extract must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The default procedure is to exclude QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in the clock.

10.3.4 Example Analytical Sequence

Refer to Attachment 1 for an example analytical sequence.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data.* This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

11.1.1 Target Analyte Identification

The judgment and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP SA-QA-008: *Evaluation of Chromatographic Data* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

11.1.2 Manual Integrations

Manual integrations must be documented in accordance with SOP SA-QA-008. Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

11.1.3 Dual Column Reporting

Refer to SOP SA-QA-008: *Evaluation of Chromatographic Data* for information on assessing and reporting data from dual columns.

11.1.4 Surrogate Evaluation

The surrogate, pentachloroethane, is spiked into each sample and QC item prior to preparation. Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Evaluate the surrogates in the same manner as the target compounds using the guidance above.

Refer to Section 11.1.5.1 for information on the surrogate dilution threshold factor.

Note: Other surrogate compounds may be used provided they produce consistent results within method-defined criteria.

11.1.5 Dilutions

If the response for an analyte exceeds the working range of the system, a dilution is required. Prepare dilutions of the extract if the dilution can be analyzed within 24 hours of the time the sample was extracted. If not, extract a smaller aliquot of sample and repeat the analysis.

Dilution from Extract

Dilution Factor	Volume of Extract	Final Volume of Dilution in Hexane
2	500uL	1000uL
5	200uL	1000uL
10	100uL	1000uL
25	40uL	1000uL
50	20uL	1000uL
100	10uL	1000uL

Dilution from Sample

Dilution Factor	Volume of Sample	Final Volume of Dilution in Water
2	25mL	50mL
5	10mL	50mL
10	5.0mL	50mL
25	2.0mL	50mL
50	1.0mL	50mL
100	0.5mL	50mL

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

11.1.5.1 Surrogate Dilution Threshold Factor

Surrogates may be diluted out if the concentration of target compounds is high or the presence of non-target compounds interferes with the quantification of the target compounds. Undetect surrogates in the sample when the dilution factor is 6 or greater. As such, recoveries must be reported as “0D”, and control limits will not apply.

An NCM must be initiated to denote this situation.

11.1.5.2 Dilutions and MS/MSD Recoveries

Matrix spike recoveries are not reported for dilutions of 6 or greater. An NCM is generated for instances where the dilution prohibits evaluation of the MS/MSD recoveries. In instances where the unspiked sample concentration is more than four times the concentration of the target compound spiked into the MS and MSD, the results are qualified with “4” or other suitable flag.

An NCM must be initiated to denote this situation.

11.1.6 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes or use the TALS Historical Date tracker feature to determine if historical data is available for review.

11.1.7 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCLs associated with this procedure are given in Attachment 6. Notify the PM immediately via a Nonconformance Memo if any sample contains a detection above these levels.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample (at the instrument)
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor

Note: This calculation assumes all applicable unit correction factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: EPA 8011specifies that the MDL must be ≤0.03ug/L.

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS) for EPA 504.1 are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits for EPA 504.1 and does not utilize in-house nor laboratory-derived limits for the evaluation of batch QC items.

The control limits for the batch QC items (LCS, MS) for EPA 8011 are not specified in the reference method; therefore, the laboratory utilizes in-house nor laboratory-derived limits for the evaluation of batch QC items for EPA 8011.

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017.

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four

consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 13 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Flammable waste (acetone, hexane, and methanol from extracts, rinsings, and standards) - Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.
- Sample residue from the sample vials contains hexane - The samples are poured into a separatory funnel that is used to separate the hexane layer from the aqueous layer. The aqueous layer is discarded down the sink, and the hexane layer is contained in a flammable waste container.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, December 1986 and February 2007.
  - Method 8011: 1,2-Dibromoethane and 1.2-Dibromo-3-Chloropropane by Microextraction and Gas Chromatography, Revision 0; July 1992
  - Method 8000B: Determinative Chromatographic Separations, Revision 2; December 1996.
- *Methods for the Determination of Organic Compounds in Drinking Water:*
  - EPA Method 504.1, 1,2-Dibromoethane (EDB), 1.2-Dibromo-3-Chloropropane (DBCP), and 1,2,3-Trichloropropan (123TCP) in Water by Microextraction and Gas Chromatography, Revision 1.1, Munch, J.W. 1995

16.0 Method Modifications and Clarifications

16.1 The reference method was written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure.

16.2 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this frequency via the Low-level LCS required by both methods.

16.3 The amount of sodium chloride added to the samples differs between EPA 504.1 (6g) and EPA 8011 (7g). This SOP directs the analyst to use 6g per sample, calibration standard, and QC item. The addition of salt to the sample is to increase the polarity of the sample matrix, which increases the tendency for the target compounds to partition into the non-polar solvent (hexane). Six grams of salt is adequate to achieve this purpose. In addition, the laboratory believes this minor modification of the method to have no impact on sample results since the samples and calibration standards are processed in the same manner.

16.4 A calibration standard at one half the routine RL of 0.020ug/L is included in the initial calibration to support the RL of 0.010ug/L required by one or more state agencies.

16.5 The laboratory has incorporated the batch QC items as outlined in Section 9.1. Some additional QC items are performed (above those required in the reference methods) to

satisfy common state regulatory and/or client requests for precision data and/or to facilitate scheduling and data evaluation. Additionally, some QC items are combined (such as the daily Low-level LCS required for the EPA Drinking Water Manual and the weekly Low-level LCS required by EPA 504.1; or for EPA 504.1, the CCV and the LCSD required for batches of 11-20) to facilitate analysis and performing both EPA 504.1 and EPA 8011 in the same batch.

The method-specified batch QC items are as follows:

EPA 504.1: Lab reagent blank and field reagent blank each day; 1 LCS per 10% of samples (70-130%R); Low-level LCS weekly (60-140%R); 1 MS per batch (65-135%).

EPA 8011: reagent and calibration blank per batch; check sample at 0.25ug/L for 5% of samples (60-140%R); QC reference sample at 0.10ug/L weekly (60-140%R); MS/MSD or sample duplicate daily.

EPA Manual for the Certification of Laboratories Analyzing Drinking Water: method blank per batch, LCS per batch, Low-Level LCS daily.

16.6 EPA 8011 does not give CCV acceptance criteria. The laboratory uses 20%D, which is consistent with the guidance given in EPA 8000C.

16.7 EPA 504.1 includes the use of Field Reagent Blanks (i.e., trip blanks). The laboratory does not normally include these in outgoing bottle kits; however, this task can be accommodated upon client request.

16.8 SW-846 does not specifically address bottle types for EPA 8011. The bottle type specified in EPA 504.1 (i.e., a 40mL VOA vial with sodium thiosulfate dechlorination agent) can be used for both EPA 504.1 and EPA 8011, or alternatively, the bottle type specified in EPA 8260 (i.e., a 40mL VOA vial with HCl preservative) or that specified in EPA 624 (i.e., a 40mL unpreserved VOA vial) can be used for this method.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Standard Preparation Recipes
Attachment 6: Maximum Contaminant Level (MCL) Table
Attachment 7: Labware Cleaning Procedures

Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary
Thirty-five milliliters of sample are extracted with two milliliters of hexane. The extract is analyzed by gas chromatography utilizing dual capillary columns and dual electron capture (EC) detectors. Calibration standards are extracted and analyzed in the same manner as the samples.

Example Analytical Sequences
Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Instrument Blank	Hexane
ICAL	Minimum of five points
Instrument Blank	Hexane
ICV	Second source standard
Instrument Blank	Hexane
Field and QC samples	Not to exceed 20 field samples
CCV	Mid-level
Instrument Blank	Hexane
Field and QC	Not to exceed 20 field samples
CCV	Mid-level calibration standard
Instrument Blank	Hexane

Analytical Sequence for samples not immediately following an initial calibration:

Description	Comments
Instrument Blank	Hexane
RL Standard	Per batch
CCV	Mid-level calibration standard
Instrument Blank	Hexane
Field and QC Samples	Not to exceed 20 field samples
CCV	Mid-level
Instrument Blank	Hexane
Field and QC Samples	Not to exceed 20 field samples
CCV	Mid-level
Instrument Blank	Hexane

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Listed below are the holding times and preservation requirements:

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Water	3 x 40mL VOA; no headspace	35mL	35mL	None	0-6°C	Sodium Thiosulfate	Extraction: 14 days from collection Analysis: 24 hours from extraction

Attachment 3: QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) - Minimum 5 points -Extracted	Analyzed initially prior to sample analysis, when major instrument maintenance performed, or when CCV fails	EPA 504.1: %RSD<20% r^2>0.990 EPA 8011: %RSD<10% r^2>0.990	Refer to SOP SA-QA-016
Initial Calibration Verification (ICV) - 2nd Source - Extracted	Analyzed after each ICAL. Note: LCS is used to satisfy since from a second source.	70-130% Recovery	Refer to SOP SA-QA-016
Continuing Calibration Verification (CCV)	Extracted each day samples are prepared. Analyzed initially, after every 20 samples (not to exceed 12 hours), and at the end of the sequence - Concentration must be varied throughout the mid-range.	EPA 504.1: <30%D EPA 8011: <20%D	Refer to SOP SA-QA-016
Calibration Blank (CCB/ICB)	After ICV and every CCV	<MDL	Terminate the analysis; correct problem; reanalyze affected samples.
Surrogate	All field, batch QC, & instrument QC samples	Within TALS MLG limits	Refer to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	Extracted together w/in 24-hr period; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	<MDL	Refer to SOP SA-QA-017
Laboratory Control Sample (LCS)	One per batch	Within limits listed in the MLG	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient volume provided for MS/MSD	Within limits listed in the MLG	Refer to SOP SA-QA-017
Low-Level Laboratory Control Sample (LLCS) / Method Detection Limit Verification (MDLV)	One per batch	60-140%R	Refer to SOP SA-QA-017
Matrix Spike (MS)	One per batch	Within limits listed in the MLG	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	One per batch	Within limits listed in the MLG	Refer to SOP SA-QA-017
Column Resolution Check (DBCM Check)	One per batch	Baseline resolution between DBCM and EDB	- Perform column maintenance - Adjust column flow/temperature to attain resolution. - Install new column or columns.

QC Item	Frequency	Criteria	Corrective Action
Retention Time Window Determination	Annually	Refer to SOP SA-QA-016	Refer to SOP SA-QA-016
Initial Demonstration of Capability (IDOC)	Initially; Per analyst / matrix / method / analyte combination	Within limits listed in the MLG	Refer to SOP SA-QA-006 (Unsupervised work must not begin without successful completion of IDOC.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Within limits listed in the MLG	Refer to SOP SA-QA-006
Method Detection Limit (MDL)	Upon method/instrument set-up	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, and quarterly thereafter	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

Refer to the instrument manufacturer's guides for trouble-shooting items.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Guard Column/Injector							X	Change sleeve and cut front of guard column, recommended daily
Septum							X	Replace, recommended daily
Splitless Disc							X	Replace, recommended daily
Autosampler							X	Syringe cleaned or replaced as needed
Column							X	Change column

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Attachment 5:
Standard Preparation Recipes

Stock Standard Mixes

Stock/Mix	TALS ID	Vendor/ Part Number	Concentration (ug/mL)
504.1 Mixture	SG504ICV_	Ultra DWM-514	200
552.2 Internal Standard (1,2,3-Trichloropropane Soln.)	SG123TCP_	Ultra PPS251-1	1000
504.1 EDB/DBCP Spike Std (Second Source)	SG504CAL_	Accustandard M-504	200
Dibromochloromethane (Stock)	SG504DBCM_	Ultra HC-100	100
504 Surrogate (Pentachloroethane)	SGPCE504_	Restek 30404	2000

Storage: <-10℃
Expiration:
Unopened: Manufacturer's expiration date
Opened: 6 months from opening

504 Intermediate Standard (TALS ID = 504 INT A)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
504.1 Mixture (SG504CAL_)	125	2.0	12.5 (EDB/DBCP)
552.2 Internal Standard (SG123TCP_)	125		62.5 (1,2,3-TCP)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504 Working Standard #1 (TALS ID = 504 WS#1)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
504 Intermediate A (504 INT A)	50	10	0.0625 (EDB/DBCP) 0.3125 (1,2,3-TCP)
504 Surrogate (504 Penta_)	500		0.025 (PCE)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504 ICV/LCS Spike Intermediate Standard (TALS ID = 504 Spike_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
504.1 EDB/DBCP Spike Std (Second Source) (SG504ICV_)	12.5	25	0.10 (EDB/DBCP)
552.2 IS (SG123TCP_)	10		0.50(123TCP)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504 Surrogate Intermediate (TALS ID: 504 Penta_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
SGPCE504_	25	100	0.50 (PCE)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504 Pentachloroethane Spiking Solution (TALS ID: 504_Surr_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
504 Penta_	500	10	0.025 (PCE)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

Dibromochloromethane Intermediate Standard (TALS ID = 504-DBCM_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
Dibromochloromethane (Stock) SG504DBCM	5.6	2	0.28

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504/8011 Calibration Standards (TALS ID: enter in prep batch)

CAL STD	Volume 504 DBCM (uL)	Volume 504 WS#1 (uL)	Volume of Reagent Water (mL)	EDB/DBCP		1,2,3-TCP		DBCM
				ug/mL[1]	ug/L[2]	ug/mL[1]	ug/L[2]	ug/mL[1]
1	5	5	35	0.000156	0.0089	0.000781	0.045	0.0175
2	0	10	35	0.000313	0.018	0.001563	0.089	0
3	0	20	35	0.000625	0.036	0.003125	0.18	0
4	0	35	35	0.001094	0.063	0.005469	0.31	0
5	0	50	35	0.001563	0.089	0.007813	0.45	0
6	0	65	35	0.002031	0.12	0.010156	0.58	0
7	0	80	35	0.0025	0.14	0.0125	0.71	0
8	0	100	35	0.0031250	0.18	0.015625	0.89	0

Storage: Not applicable; made fresh each day
Expiration: 24 hours

(1) Concentration in extract, final volume = 2.0mL
(2) Concentration in standard, initial volume = 35mL

504/8011 Initial Calibration Verification/LCS (TALS ID: enter in prep batch)*

STD	Volume 504 Spike (uL)	Volume 504 Surr DL (uL)	Volume of Reagent Water (mL)	Final Concentration (EDB/DBCP) (ug/L)	Final Concentration (1,2,3-TCP) (ug/L)
ICV/LCS	35	35	35	0.10	0.50

Storage: Not applicable; made fresh each day
Expiration: 24 hours
*Also used to prepare the MS and MSD

504/8011 Column Resolution/RL Check (TALS ID: enter in prep batch)

STD	Volume of Intermediate (uL)		Volume of Water	Concentration of RL/Resolution Check (ug/L)		
	504 INT A	504-DBCM		EDB/DBCP	1,2,3-TCP	DBCM
RL/ResCheck	5	5	35mL	0.010	0.050	1.0

Storage: Not applicable; made fresh each day
Expiration: 24 hours

Guidance for Preparing Intermediate and Working Standards in Methanol

- Clean and rinse volumetric flask with methanol.
- Add methanol to volumetric flask to approximately one half volume.
- Add standard to volumetric flask, inserting the syringe needle under the surface of the methanol.
- Dilute to volume with methanol, cap, and invert three times to mix.
- Transfer by gently pouring the newly made standard into a labeled storage with minimal headspace and seal with Teflon-lined screw or crimp cap.
- Store at -10C in freezer

Guidance for Preparing Calibration and Verification Standards in Water

- Add 35mL of reagent water to a 40mL VOA vial.
- Add standard to the vial, inserting the syringe needle under the surface of the water.
- Cap the vial and mix by inverting three times.
- Use immediately.

Attachment 6:
Maximum Contaminant Level (MCL) Table

Primary Drinking Water Regulations		
Contaminant	**MCL (mg/L)**	**MCL (ug/L)**
1,2-Dibromo-3-chloropropane (DBCP)	0.0002	0.2
Ethylene Dibromide (EDB)	0.00005	0.05

Attachment 7: Labware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES

SEMIVOLATILE GC LAB

1. Scrub with tap water and Liquinox.
2. Rinse 3 times thoroughly with tap water.
3. Rinse 3 times thoroughly with Acetone.
4. Place volumetric top-down within storage rack and allow to air dry.
5. Store in closed drawer.

FSG046:12.04.13:0

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18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Added Volumetric Container Table. Section 6.2
- Adjusted sample collection and storage conditions to reflect 0-6°C. Section 8.1
- Removed requirement to extract an ICAL and ICV with each batch of samples. Once the ICAL is established, subsequent CCVs must be extracted each day samples are extracted. Section 9.2.2 and Attachment 3
- Changed CCV criteria for EPA 8011 from 15%D to 20%D. Section 9.2.5, Section 16.6, and Attachment 3
- Revised standard preparation instructions, concentrations, and nomenclature to reflect current laboratory practice. Attachment 5
- Added Labware Cleaning Procedures. Attachment 7

VOLATILE COMPOUNDS IN DRINKING WATER BY GC/MS

(Methods: EPA 524.2)

Approvals (Signature/Date):

Andrea Teal 12/04/2015
Andrea Teal Date
Quality Assurance Manager

Whitney Palefsky 12/04/2015
Whitney Palefsky Date
Environmental Health & Safety Coordinator

Charlton A. Riegner 12/04/2015
Charlton Riegner Date
Technical Manager

1.0 Scope and Application

This SOP gives the procedures for the determination of volatile organic compounds in water samples by gas chromatography/mass spectrometry (GC/MS).

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOC onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

This SOP is based on the following method: EPA 524.2.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

THM (Trihalomethanes) - The four THM are chloroform, dichlorobromomethane, dibromochloromethane, and bromoform.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions (Attachment 8) to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Methanol must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 VOC commonly used in the laboratory may be a major source of contamination. Hexane, methylene chloride, acetone, freon, 2-butanone (MEK), toluene, and isopropanol are all common laboratory solvents and tend to cause the most interference. The analyses of highly concentrated samples (>1ppm) may also affect the succeeding runs. “Carryover” can occur when low concentration samples are analyzed after high level samples. Reagent blanks must be analyzed periodically to check for laboratory contamination and carryover. The VOC laboratory must be kept as free from contaminants as possible.

4.2.4 Samples containing chlorine must be treated with ascorbic acid. If excess chlorine is not destroyed, the concentration of some compounds formed when water is chlorinated (for example, trihalomethanes) may not reflect the analyte concentration at the time of sampling. Samples for trihalomethanes are dechlorinated using sodium thiosulfate.

4.2.5 Samples must be acidified, except samples where only trihalomethanes are requested, at the time of collection (after dechlorination) to prevent biological degradation of some VOC. The addition of acid also minimizes dehydrohalogenation of some chlorinated alkanes.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye

protection (safety glasses or face shield), disposable nitrile gloves (or equivalent), and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Hydrochloric acid is extremely hazardous as an oxidizer, a corrosive, and a poison, and it is reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and may lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage. Concentrated acids should be used in a fully functional fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP.

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the “MSDS” link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hydrochloric Acid	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Analytical Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Agilent (HP) 5973 Mass spectrometer equipped with a capillary direct interface.

Agilent (HP) 6890 Gas chromatograph with spilt/splitless injector. The exit vent must have a carbon trap in-line to collect the volatile compounds that are vented during the transfer from the purge and trap device. The carbon traps should be changed a minimum of every three months.

Restek RTX-624 Column: 20m x 0.18mm ID, 1.0um film thickness, or equivalent.

EST Encon purge and trap concentrator with 5mL sparge vessel, or equivalent.

EST Centurion Autosampler, or equivalent.

Supelco Vocarb 3000 trap or equivalent. Other traps may be used as long as the target compounds can be detected at the required quantitation limit and the IDOC requirements are met.

6.2 Analytical Data System / Software / Hardware

Chemstation software is used on a Windows-based PC to schedule and acquire data. CHROM software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's LIMS system (i.e., TALS). CHROM software has the capability of processing stored GC data by recognizing a GC peak within any given retention time window and comparing the retention time of the sample to the retention times of the standards analyzed under the same conditions. The software also allows calculation integration of the peak responses, response factors, construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

6.3 Volumetric Labware

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*. Refer to Attachment 8 for labware cleaning procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Verification Criteria
Volumetric Flasks (Class A)	25mL 50mL	QUANTITATIVE	Preparing Standards; Preparing Dilutions	None	None (Purchased Class A)
VOA Vials	40mL	QUANTITATIVE	Sample Collection and Analysis	Per Lot	Accuracy = 2% Precision = 1%
Gas-Tight Syringes	Various	QUANTITATIVE	Standard Preparation and Spiking	None	None (If received with COA)
Mini-Nert Vials	Various	Qualitative	Standard Storage	None	None

6.4 Lab Supplies

pH paper

Residual Chlorine Check Strips – starch iodide strips; provide a quick and easy way to verify if the sample was dechlorinated properly. Store in original, capped container and use within the manufacturer's expiration date.

Detergent – Liquinox used for washing non-disposable labware.

6.5 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are packaged in a kit which contains:

- 3x 40mL VOA vials containing ascorbic acid granules (used for dechlorination)
- 1 HCl dropper containing 1mL 1:1 HCl (used for preservation)

The kits are purchased with a Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

Unless listed elsewhere in this SOP, the expiration dates given below apply.

7.1.1 The expiration date for unopened standards and reagents is the manufacturer's expiration date.

7.1.2 The expiration date for opened stock reagents is the manufacturer's expiration date or 5 years from the date opened, whichever is sooner.

7.1.3 The expiration date for opened stock standards is the manufacturer's expiration date or 1 month from the date opened, whichever is sooner.

7.1.4 The expiration date for prepared reagents is 6 months from the date prepared or the expiration date of the parent reagent, whichever is sooner.

7.1.5 The expiration date for prepared standards is 1 month from the date prepared or the expiration date of the parent standard, whichever is sooner.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Methanol must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Laboratory Reagent Water – ASTM Type II, obtained from on-site well

Methanol – Purge and Trap grade
Storage: Flammable Cabinet

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached to the standard in TALS.

Refer to Attachment 6 for standard preparation information.

Unopened source standards must be stored at manufacturer's recommended conditions. After opening, standards are stored in the freezer at < -10°C.

8.0 Sample Collection, Preservation, Shipment, and Storage

Aqueous samples are routinely collected in triplicate. Two vials are retained for analysis and the third vial is used to check the sample pH and for the presence of residual chlorine. This "sacrifice" vial should not be used for analysis unless all other vials have been consumed. If the "screening vial" is used for analysis, a Nonconformance Memo (NCM) must be initiated.

Samples are routinely collected with no headspace in 40mL vials equipped with Teflon-lined caps. The samples are dechlorinated with 25mg of ascorbic acid and acidified with about 1.0mL of 1:1 HCl per 40mL of sample at the time of collection. The preservative should be sufficient to achieve a sample pH of less than 2. The dechlorination agent should be sufficient to remove residual chlorine from the sample.

Samples must be iced at the time of collection and refrigerated at 0-6°C (less than 6°C with no frozen samples) in the lab until analysis. Samples must be analyzed within 14 days of collection. If the samples are unpreserved or if the pH >2, the samples must be analyzed within 24 hours of collection.

Note: If Total Trihalomethanes (THM) are the only analytes requested, the acid may be omitted and the samples may be dechlorinated with 4mg of sodium thiosulfate per 40mL of sample at the time of collection.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives and/or de-chlorination agents. NCMs must be initiated for samples that are received containing headspace.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatile Samples* for additional information.

8.1 Preservation Checks

These checks can be performed upon receipt or prior to preparation.

8.1.1 Mix the sample by inverting. Using a 100uL syringe, withdraw approximately 50uL of sample through the septa.

8.1.2 Dispense a small amount of sample onto a piece of narrow range pH paper and note the pH. Record the pH in the PRESERV_CHK method in TALS.

If the pH is greater than or equal to 2, initiate an NCM noting that the pH was outside of the preservation requirements.

Note: If the pH is greater than or equal to 2, a 24-hour holding time is enacted. Notify the Project Manager via a NCM if the 24-hour holding time is not met.

8.1.3 Dispense remaining sample onto a piece of starch iodide paper and note the color change of the paper.

If the paper turns blue or black, residual chlorine is present. Record whether the sample contains residual chlorine in the PRESERV_CHK method in TALS. Initiate a Nonconformance Memo if the sample contains residual chlorine.

8.1.4 Repeat steps 8.1.1 through 8.1.3 individually for each sample. Check the pH, then record the pH result in the TALS batch; check for residual chlorine, then record the residual chlorine result in the TALS batch – prior to proceeding to the next sample.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An analytical batch consists of up to 20 environmental samples and the associated QC items analyzed together within a 12 hour period. The minimum QC items required for each batch are: a method blank, a laboratory control sample (LCS), a low-level LCS (spiked at the reporting limit), and a matrix spike (MS), and a matrix spike duplicate (MSD)

If there is insufficient sample to perform the required MS and/or MSD, the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample is defined as receiving less than 4 vials.

Note: The LCS must be analyzed in duplicate at least once a quarter.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Note: The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. Therefore, if analyzing drinking water samples by EPA 524.2, an LCS at the RL must also be included in the required batch QC.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Instrument QC

The term “clock time” or “analytical clock” refers to the amount of time that can pass before additional instrument QC items must be performed. The analytical clock begins with the injection of the BFB, and all subsequent injections must be completed before the

clock time expires – at which point new instrument QC is performed and a new clock is initiated.

The clock time for EPA 524.2 is defined as 12 hours.

Note: Due to instrument configurations employing dual concentrators, most of the laboratory instruments can analyze more than 20 injections within the designated clock times. An analytical batch is still defined as 20 field samples; therefore, if more than 20 field samples are analyzed within a clock, additional batch QC is required (i.e., another method blank, LCS, and MS/MSD must be performed).

9.2.1 Tune Check

Inject 1uL of the 25ng/uL BFB standard.

Note: The analysis may be performed using purge and trap or by direct injection of the BFB standard. Mass spectrometer conditions must be the same as for the standard and sample analyses. The temperature programs may be different to allow for timely elution of BFB.

Evaluate the spectrum of the BFB peak. Test the apex of the peak first against the acceptance criteria. If the apex does not meet the criteria, evaluate the scans plus one and minus one scan from the apex. An average spectrum across the peak may also be evaluated against the criteria. If background subtraction is required, choose a spectrum at least ten scans before the elution of the peak for background.

TUNING AND MASS CALIBRATION ACCEPTANCE CRITERIA	
m/e	**Abundance Criteria**
50	15-40% of mass 95
75	30-80% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	< 2% of mass 174
174	Greater than 50% of mass 95
175	5-9% of mass 174
176	> 95% but < 101% of mass 174
177	5-9% of mass 176

Note: The p-BFB analysis must meet the criteria before any standards or samples may be analyzed. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. If there is any question about whether the BFB passes the criteria, contact the supervisor immediately before proceeding.

If the p-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the p-

BFB analysis, other corrective measures may include remaking the p-BFB standard, cleaning the instrument ion source, etc. Additionally, the chromatogram of the tuning analysis should be checked for acceptable baseline and the p-BFB peak should be symmetrical.

9.2.2 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 3 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

Standard Level	Concentration (ug/L)
1	0.5
2	1.0
3	2.0
4	5.0
5	10
6	20
7	50
8*	100

*Used for TTHMs only.

Refer to Attachment 6 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

Note: EPA 524.2 requires a minimum of a 3-point calibration curve for a 20 fold concentration range, a 4-point calibration curve for a 50 fold concentration range, and a 5-point calibration curve for a 100 fold concentration range.

9.2.2.1 ICAL Criteria

The relative standard deviation of the calibration standards must be <20% for the initial calibration curve to be acceptable.

If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted.

9.2.3 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The ICV must be within 30% to be acceptable.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to level 6 of the ICAL. Refer to Attachment 6 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

Note: The LCS may be used to satisfy the ICV requirement if it is prepared from a second source and meets the criteria outlined above.

9.2.4 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately following the initial calibration. Continuing calibration blanks are analyzed immediately following the continuing calibration verification (CCV).

Initial and continuing calibration blanks must be <1/2RL to be acceptable.

9.2.5 Continuing Calibration Verification

The initial calibration curve must be verified at the beginning of each clock with a mid-level standard.

The CCV must be within 30% to be acceptable.

The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to level 6 of the ICAL. Refer to Attachment 6 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.6 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. Fluorobenzene is the internal standard.

Prior to analysis, this internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. A concentration of 10ug/L is used.

The response of the ISTD in the ICV/CCV must be within 30% of the response of the ISTD in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

The response of the ISTD in the samples and batch QC items must be within 30% of the response of the previous CCV. If the response is outside of this range, corrective action must be taken.

9.2.7 Surrogate

This procedure uses surrogates to evaluate the analytical process. 1,2-Dichlorobenzene-d4 and 4-Bromofluorobenzene are the surrogates.

Prior to analysis, this surrogate is added to all samples and QC items. The concentration of the surrogate is the same in all field samples and QC samples. A concentration of 10ug/L is used.

The percent recovery of the surrogate in all field samples and QC samples must be within the limits listed in the Method Limit Groups (MLGs) in TALS. If the percent recovery is outside of this range, the analysis of the sample must be repeated. Repeated failure of the surrogate percent recovery may indicate instrumentation problems.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

Composite samples can be prepared using the guidance provided in SOP SA-QA-015: *Compositing, Homogenization, and Segregation of Samples*.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for additional information.

10.2 QC Sample Preparation

10.2.1 Method Blank – The method blank is prepared as follows: Fill a 50mL volumetric with reagent water. Add 50uL of ISSU. Invert flask three times and transfer contents to a 40mL VOA vial (containing HCl preservative) with no headspace. Place on instrument to be analyzed.

10.2.2 Laboratory Control Sample – The LCS is prepared as follows: Fill a 50mL volumetric with reagent water. Add 20uL of Mega Mix, 20uL of Additional Mix, 50uL of ISSU, and 160uL

of MeOH. Invert flask three times and transfer contents to a 40mL VOA vial (containing HCl preservative) with no headspace. Place on instrument to be analyzed.

10.2.3 Low-Level Laboratory Control Sample – The LLCS is prepared as follows: Fill a 50mL volumetric with reagent water. Add 0.5uL of Mega Mix, 0.5uL of Additional Mix, 50uL of ISSU, and 199uL of MeOH. Invert flask three times and transfer contents to a 40mL VOA vial (containing HCl preservative) with no headspace. Place on instrument to be analyzed.

10.2.4 Matrix Spike – Matrix spikes are prepared as follows: Spike 17.2uL of Mega Mix, 17.2uL of Additionals Mix, 43uL of ISSU, and 137.6uL of MeOH into a 40mL VOA vial containing the sample designated for the MS and MSD. Place in the instrument to be analyzed.

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Note: The drinking water methods are prescriptive. For this reason, items such as purge volume, purge gas, purge time, carrier gas, etc. must match the EPA method.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

Note that the MS must be set to monitor ions between 35 and 260amu with a scan rate of 1 second or less. The purge time must be 11 minutes. All other parameters may be changed to optimize the system.

Column: Restek RTX-624 0.18mm x 20m x 1.0um, or equivalent

Helium carrier gas flow rate: 0.5mL/min (constant flow)
Inlet Pressure: 15.8 psi
Total Flow: 28.2mL/minute
Split Ratio: 50:1 (Routine and Tune Check)
Split Ratio: 25:1 (UCMR List 1 Compounds)
Split Flow: 25mL/min
Gas Saver: 20.0 mL/min @ 2.00 min

Routine Targets and UCMR List 1	**BFB Tune Check**
Initial column temperature: 40°C for 1min	Initial column temperature: 50°C for 1min
Column temperature program 1: 17°C/min	Column temperature program 1: 17°C/min
Final column temperature: 200°C for 7min	Final column temperature: 200°C for 7min
Run Time: 11.41 minutes	Run time: 8.82 minutes

Injector temperature: 250°C
Mass range: 35-260amu
Solvent Delay: 0.90 Minutes
Threshold: 150 Sample #: 2 A/D Samples: 4
MS Quad Temperature: 150°C MS Source Temperature: 250°C
EM Absolute: TRUE
Resulting EM Voltage: EM voltage set at AUTOTUNE + 200
Tune File: TUS.U for instrument MSS
Tune File: TUU.U for instrument MSU

Purge and Trap Instrument Conditions

Purge Time: 11 min
Purge Temperature: Ambient
Desorb Time: 0.5 min
Desorb Temperature: 250°C
Bake Time: 8 min at 260°C
Purge Flow: Approximately 35 mL/min. Adjust to maximize response of chloromethane and bromoform.
Valve Temperatures: 150°C
Transfer Line: 150°C

10.3.2 Internal Standard (ISTD)

Prior to analysis, 43uL of ISSU must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. A concentration of 10ug/L is used.

10.3.3 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.2. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.3, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5.

10.3.4 Sample Analysis

Remove the samples from the refrigerator and allow them to come to room temperature.

The sample must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The default procedure is to exclude QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in the clock.

10.3.5 Example Analytical Sequence

See Attachment 1 for an example analytical sequence.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data.* This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data review and reporting must be performed in accordance with SA-QA-002: *Data Generation and Review.*

11.1 Qualitative Analysis of Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1 Two criteria must be met in order to identify a target-compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{retention\ time\ of\ the\ target\ compound}{retention\ time\ of\ the\ associated\ internal\ standard}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. These ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.1.5 MS/MSD Evaluation

If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data is flagged.

11.1.2 Evaluation of Tentatively Identified Compounds (TICs)

Refer to Attachment 11 of SOP SA-QA-008: *Evaluation of Chromatographic Data* for the laboratory's TIC processing procedures.

11.1.3 Dilutions

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

11.1.4 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.1.5 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met, the Department Supervisor must be contacted immediately.

Benzene, toluene, ethylbenzene, and the xylenes are generally present together in samples and indicate the presence of gasoline

m/p-Xylenes are generally higher than o-xylene

Hydrocarbons present is samples containing gasoline generally contain mass 43 and may co-elute with target analytes with mass 43 as the quant or confirmation ion or may skew the spectrum of a compound with mass 43 as part of the spectrum.

Cis- isomers are generally more prevalent than the trans- isomers

Pay particular attention to the retention time of isomer because the only way to positively identify them is by retention time. The isomers are:

1,1-dichloroethane and 1,2-dichloroethane
1,1-dichloroethene, cis-1,2-dichloroethene, and trans-1,2-dichloroethene
1,1,1-trichloroethane and 1,1,2-trichloroethane
ethyl benzene, m/p-xylene, and o-xylene
1,3-dichlorobenzene, 1,4-dichlorobenzene, and 1,2-dichlorobenzene
1,1-dichloropropene, cis-1,2-dichloropropene, and trans-1,2-dichloropropene
2-chlorotoluene and 4-chlorotoluene
1,2,3-trichlorobenzene and 1,2,4-trichlorobenzene
1.3.5-trimethylbenzene and 1,2,4-trimethylbenzene
4-methyl-2-pentanone (MIBK) and 2-hexanone
n-butylbenzene, sec-butylbenzene, tert-butylbenzene, and isopropylbenzene

Higher chlorinated alkanes and alkenes may have lower chlorinated alkanes or alkenes present due to degradation. The following table lists some common chlorinated compounds and their degradation products. Look for the degradation product(s) when the concentration of the compound in the left column is present at high concentrations.

Analyte	Degration Product
1,1,2,2-tetrachloroethane	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) vinyl chloride 1,1,2-trichloroethane (1,1,2-TCA) 1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,2-trichloroethane (1,1,2-TCA)	1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,1-trichloroethane (1,1,1-TCA)	1,1-dichloroethene (1,1-DCE) 1,1-dichloroethane (1,1-DCA) Chloroethane
Carbon tetrachloride	Chloroform Methylene chloride Chloromethane
Tetrachloroethene (PCE) (PCE = perchloroethylene which is a common name for tetrachloroethene)	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) Chloroethene
1,2,4-trichlorobenzene	1,4-dichlorobenzene (1,4-DCB) 1,2-dichlorobenzene (1,2-DCB) Chlorobenzene

Trihalomethanes are formed when water from a natural source (river, well, etc.) is chlorinated. Usually, THM will be present in the relative concentrations as follows:
chloroform >> dichlorobromomethane > dibromochloromethane >> bromoform.

11.1.6 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCLs associated with this procedure are given in Attachment 8. Notify the PM immediately via a Nonconformance Memo if any sample contains a detection above these levels.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor

Note: This calculation assumes all applicable unit correction factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

In addition to the requirements in SOP SA-QA-007, EPA 524.2 also requires that MDL studies be performed over multiple days.

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Note: The IDOC must meet 80-120% recovery and less than 20% RSD.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess reagents – Dispose as outlined below.

Material	Treatment	Disposal Destination
Methanol	None	Flammable Waste Drum
Standards	None	Flammable Waste Drum
Hydrochloric Acid and Solutions	Neutralize	Sink

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- SOP SA-VO-001*: Preparation, Storage, and Screening of Volatiles Samples*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- US EPA 524.2 Revision 4.1: *Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry*; 1995

16.0 Method Modifications

16.1 The reference method was written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure.

16.2 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each batch of samples. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory defaults to 50-150%. These criteria are required for THMs, as specified in the Disinfection By-Product Rule.

16.3 The laboratory has incorporated the minimum batch QC items as outlined in Section 9.1. Some additional QC items are routinely performed above those required in the EPA 524.2 reference method (i.e., MS/MSD and/or LCS/LCSD) to satisfy common regulatory and/or client requests for precision data and/or to facilitate scheduling and data evaluation.

16.4 Due to the volatile nature of the analytes tested, the laboratory sacrifices a vial to be used for pH check, residual chlorine verification, and screening. The laboratory applies the pH and residual chlorine values identified on this vial to the remaining vials submitted for that sample (e.g., if the pH of the tested vial is acceptable, the remaining vials for that sample are assumed to be acceptable). The practice of checking pH prior to analysis allows for re-adjustment of holding times based on the preservation of the sample, as outlined in Attachment 2.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Standard Preparation
Attachment 6: List of Regulated Analytes and MCLs
Attachment 7: Quant Ions
Attachment 8: Glassware Cleaning Procedures

Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary

Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Analytical Sequence

Description	Comments
Blank	
Tune	12-hour clock begins with injection of the tune
Initial Calibration	
ICV	Second Source
ICB	
Samples & Batch QC Items	Not to exceed 12 hours; last injection must occur before 12 hours from BFB injection
Tune	12-hour clock begins with injection of the tune
CCV	20ug/L
CCB	
Samples & Batch QC Items	Not to exceed 12 hours; last injection must occur before 12 hours from BFB injection
Tune	12-hour clock begins with injection of the tune
CCV	20ug/L
CCB	

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation[1]	Thermal Preservation	Holding Time[3]
Water	3 x 40mL VOA vial	40mL	40mL	Ascorbic Acid	1:1 HCl	0-6°C[2]	pH<2: 14 days pH>2: 24 hours
Water (TTHM Only)	3 x 40mL VOA vial	40mL	40mL	Sodium Thiosulfate	Not Applicable	0-6°C[2]	14 days

[1]Samples must be dechlorinated prior to acidification.
[2]Samples are collected on ice and maintained at <6°C with no frozen samples.
[3]Holding time is from sample collection to analysis.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Clock Time	12 hours	Clock time starts with the injection of the BFB. Analysis of samples and QC items must conclude within expiration of clock time. Subsequent analysis requires new BFB.	Not applicable
Tune Standard (BFB)	At beginning of each clock	Refer to Section 9.2.1.	- Perform instrument maintenance - Re-tune.
Initial Calibration (ICAL) - Minimum 3 points	Upon instrument set-up, and after unsuccessful CCV	%RSD < 20% If %RSD > 20%, use curve fit w/ r^2>0.990.	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Initial Calibration Verification (ICV) - Second Source	After each ICAL	%RSD < 30%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Continuing Calibration Verification (CCV)	After BFB	%RSD < 30%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate

QC Item	Frequency	Criteria	Corrective Action
Calibration Blank (ICB/CCB)	After ICV and every CCV	<1/2RL	Refer to SOP SA-QA-017
Internal Standards (ISTD)	Spiked in all CCVIS, samples, and batch QC items	CCVIS: - Area within 30% of CCV in ICAL. Samples & batch QC items: - Area within 30% of previous CCVIS.	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	Spiked in all samples and batch QC items.	70-130%	-Evaluate chromatogram, spectra, and integrations -Reanalyze sample, if sufficient sample available
Analytical Batch Definition	Analyzed together w/in 12-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per analytical batch	<1/2RL	Refer to SOP SA-QA-017
Laboratory Control Sample (LCS)	One per analytical batch	70-130% Rec	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per analytical batch, when insufficient sample is provided for MS/MSD	70-130% Rec; <30%RPD	Refer to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
Low-Level Laboratory Control Sample (LLCS)	One per analytical batch	50-150% Rec	Refer to SOP SA-QA-017
Matrix Spike (MS)	One per analytical batch (If additional sample volume provided by client.)	70-130% Rec	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	One per analytical batch (If additional sample volume provided by client.)	70-130% Rec; <30%RPD	Refer to SOP SA-QA-017
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	80-120% Rec; <20% RPD	Refer to SOP SA-QA-006 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL) - Must be performed over multiple days	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

QC Item	Frequency	Criteria	Corrective Action
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Injector Port							X	Replace septum, sleeve, inlet seal, and washer (Recommend every 2 weeks)
Sparge Tubes							X	Clean (Recommend every 3 months)
Column							X	Change column (Recommend annually)

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation,

the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Attachment 5:
Standard Preparation

Purchased Standards

Mega Mix, 2000 ug/mL – NSI Solutions

Mega Mix 2 (Secondary Standard), 2000ug/mL – Restek

Gases Mix, 2000ug/mL – Supelco

Gases Mix 2 (Secondary Standard), 2000ug/mL – NSI

California Oxygenates Mix 1, 2000-10000 ug/mL – Restek

California Oxygenates Mix 2 (Secondary Standard), 2000-10000 ug/mL – O2Si

Volatile Organics Calibration Mix, 5000ug/mL – Restek

Ketone 2 (Secondary Standard) 2000ug/mL – Supelco

Freon, 2000ug/mL – O2SI

Freon 2 (Secondary Standard), 2000ug/mL – Ultra Scientific

Internal Standard and Surrogate Mix, 2000 ug/mL

BFB (Tune) - NSI

Prepared Standards

524 Mega Mix (Working Standard), 50-150ug/mL – Prepared by adding 250uL of Mega Mix and 250uL of Gases Mix to 10mL of methanol.

524 Mega Mix 2 (Secondary Working Standard), 50-150ug/mL – Prepared by adding 250uL of Mega Mix 2 and 250uL of Gases Mix 2 to 10mL of Methanol.

524 Additions (Working Standard), 40-200ug/mL – Prepared by adding 200uL of California Oxygenates Mix 1, 200uL of Freon, and 200uL of Volatile Organics Calibration Mix to 10mL of methanol.

524 Additions 2 (Secondary Working Standard), 40-200ug/mL – Prepared by adding 200uL of California Oxygenates Mix 2, 200uL of Freon 2, and 500uL of Ketones 2 to 10mL of methanol.

524 ISSU (Working Standard), 10ug/mL – Prepared by adding 125uL of Internal Standard and Surrogate Mix to 25mL of methanol

BFB (tune), 25ng/uL – Prepared by adding 125uL of BFB to 10mL of methanol.

ICAL Standards

Stock/Mix	1	2	3	4	5	6	7	8
	Aliquot to prepare CAL standard (uL)							
524 Mega Mix	0.5	1.0	2.0	5.0	10	20	50	100
524 Additionals	0.5	1.0	2.0	5.0	10	20	50	100
524 ISSU	50	50	50	50	50	50	50	50
Methanol	199	198	196	190	180	160	100	---
Volume of water (mL)	50	50	50	50	50	50	50	50
Concentration								
Target Compounds (ng)	2.5	5	10	25	50	100	250	500
Internal Standards (ng)	50	50	50	50	50	50	50	50

Note: All initial and continuing calibration standards are prepared in 50mL volumetric flasks and then poured into 40mL VOA vials, containing HCl preservative, for analysis on the instrument.

Attachment 6:
List of Regulated Analytes and MCLs

Analyte	MCL (ug/L)
Benzene	5
Carbon tetrachloride	5
Chlorobenzene	100
1,2-Dichlorobenzene	600
1,4-Dichlorobenzene	75
1,2-Dichloroethane	5
1,1-Dichloroethene	7
Cis-1,2-Dichloroethene	70
Trans-1,2-Dichloroethene	100
1,2-Dichloropropane	5
Ethylbenzene	700
Methylene chloride	5
Styrene	100
Tetrachloroethene	5
Toluene	1000
1,2,4-Trichlorobenzene	70
1,1,1-Trichloroethane	200
1,1,2-Trichloroethane	5
Trichloroethene	5
Vinyl chloride	2
Total Xylenes (Sum of o-xylenes and m/p-Xylenes)	10000
Trihalomethanes, total (Sum of chloroform, bromoform, dibromochloromethane, and dibromochloromethane)	100

Attachment 7:
Quant Ions

Compound	CAS	ISTD	Quant Ion	Secondary Ions	
1,1,1,2-Tetrachloroethane	630-20-6	1	131	133	119
1,1,1-Trichloroethane	71-55-6	1	97	99	61
1,1,2,2-Tetrachloroethane	79-34-5	1	83	85	168
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	1	101	51	43
1,1,2-Trichloroethane	79-00-5	1	97	83	99
1,1-Dichloroethane	75-34-3	1	63	65	83
1,1-Dichloroethene	75-35-4	1	61	96	98
1,1-Dichloropropene	563-58-6	1	75	110	77
1,2,3-Trichlorobenzene	87-61-6	1	180	182	109
1,2,3-Trichloropropane	96-18-4	1	110	112	
1,2,4-Trichlorobenzene	120-82-1	1	180	182	145
1,2,4-Trimethylbenzene	95-63-6	1	105	120	77
1,2-Dibromo-3-Chloropropane	96-12-8	1	75	157	155
1,2-Dichlorobenzene	95-50-1	1	146	148	111
1,2-Dichlorobenzene-d4 (Surrogate)	2199-69-1	1	152	115	150
1,2-Dichloroethane	107-06-2	1	62	49	64
1,2-Dichloropropane	78-87-5	1	63	76	65
1,3,5-Trimethylbenzene	108-67-8	1	105	120	77
1,3-Dichlorobenzene	541-73-1	1	146	148	111
1,3-Dichloropropane	142-28-9	1	76	78	41
1,4-Dichlorobenzene	106-46-7	1	146	148	111
2,2-Dichloropropane	594-20-7	1	77	41	
2-Butanone (MEK)	78-93-3	1	43	72	
2-Chlorotoluene	95-49-8	1	91	126	63
2-Hexanone	591-78-6	1	43	85	100
2-Methyl-2-propanol (TBA)	75-65-0	1	59	41	43
4-Bromofluorobenzene (Surrogate)	460-00-4	1	95	174	176
4-Chlorotoluene	106-43-4	1	91	126	63
4-Isopropyltoluene	99-878-6	1	119	134	91
4-Methyl-2-pentanone (MIBK)	108-10-1	1	43	85	100
Acetone	67-64-1	1	43	58	
Benzene	71-43-2	1	78	50	51
Bromobenzene	108-86-1	1	77	156	158
Bromoform	75-25-2	1	173	171	174
Bromomethane	74-83-9	1	94	96	79
Carbon tetrachloride	56-23-5	1	117	119	121
Chlorobenzene	108-90-7	1	112	77	51
Chlorobromomethane	74-97-5	1	49	130	

Compound	CAS	ISTD	Quant Ion	Secondary Ions	
Chlorodibromomethane	124-48-1	1	129	127	131
Chloroethane	75-00-3	1	64	66	
Chloroform	67-66-3	1	83	85	47
Chloromethane	74-87-3	1	50	52	
cis-1,2-Dichloroethene	156-59-2	1	61	96	98
cis-1,3-Dichloropropene	10061-01-5	1	75	77	110
Dibromomethane	74-95-3	1	93	174	95
Dichlorobromomethane	75-27-4	1	83	85	129
Dichlorodifluoromethane	75-71-8	1	85	87	101
Ethylbenzene	100-41-4	1	91	106	51
Ethylene Dibromide	106-93-4	1	107	109	
Fluorobenzene (Internal Standard)	17060-07-0	1	96	70	50
Hexachlorobutadiene	87-68-3	1	225	223	190
Isopropyl ether	108-20-3	1	45	59	87
Isopropylbenzene	98-82-8	1	105	120	77
Methyl tert-butyl ether	1634-04-4	1	73	57	
Methylene Chloride	75-09-2	1	49	84	86
m-Xylene & p-Xylene	136777-61-2	1	91	106	77
Naphthalene	91-20-3	1	128	102	51
n-Butylbenzene	104-51-8	1	91	92	134
Nitrobenzene	98-95-3	1	96	70	50
N-Propylbenzene	103-65-1	1	91	120	65
o-Xylene	95-47-6	1	91	106	77
sec-Butylbenzene	135-98-8	1	105	134	91
Styrene	100-42-5	1	104	78	103
Tert-amyl methyl ether	994-05-8	1	73	43	87
Tert-butyl ethyl ether	637-92-3	1	59	87	57
tert-Butylbenzene	98-06-6	1	119	91	134
Tetrachloroethene	127-18-4	1	166	164	168
Toluene	108-88-3	1	91	92	65
trans-1,2-Dichloroethene	156-60-5	1	61	96	98
trans-1,3-Dichloropropene	10061-02-6	1	75	77	110
Trichloroethene	79-01-6	1	130	95	132
Trichlorofluoromethane	75-69-4	1	101	103	105
Vinyl chloride	75-01-4	1	62	64	

Attachment 8: Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES

VOLATILES DEPARTMENT

1. Rinse glassware 3 times thoroughly with DI water.
2. Place glassware, top-down, within storage rack and allow to air dry.
3. If glassware was used to prepare waste sample, use FL-70 and water to scrub glassware and follow previous steps.

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18.0 Revision History

Summary of Changes from Previous Revision:

- Minor grammatical and/or editorial edits.
- Clarified section on sample collection containers to include reference to kit containing ascorbic acid and HCl. Section 6.5
- Added requirement to pour prepared method blank, LCS/LCSD, and LLCS into VOA vials containing HCl. This change stems from a 2015 MA DEP Data Review. Section 10.2.3
- Added requirement to pour prepared initial and continuing calibration standards into VOA vials containing HCL. This change stems from a 2015 MA DEP Data Review. Attachment 5

ALS Environmental

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QUALITY ASSURANCE MANUAL

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QUALITY ASSURANCE MANUAL

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14	5.9	2/5.9
21	5.10	2/5.10

1) Introduction and Scope

The purpose of this Quality Assurance Manual is to outline the quality system for the Simi Valley location of ALS Environmental (ALS Group USA Corp. dba ALS Environmental). ALS Environmental is a professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, air, and other material. Refer to Appendix J for a list of analytical capabilities specific to the Simi Valley location and corresponding accreditation status.

Quality Control (QC) procedures are used to continually assess performance of the laboratory and quality systems. ALS Environmental maintains control of analytical results by adhering to written standard operating procedures (SOPs), using analytical control parameters with all analyses, and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data. Appendix H includes a list of data qualifiers and acronyms.

This QAM is applicable to the facility listed on the title page and the off-site extraction facility located at 2360 Shasta Way, Unit G, Simi Valley California.

The information in this QAM has been organized according to requirements found in the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards (2003 and 2009), the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, USEPA, 2001; and *General Requirements for the Competence of Testing and Calibration Laboratories*, ISO/IEC 17025:2005.

2) Organization

2.1 Laboratory Organizational Structure

ALS Environmental – Simi Valley is legally identifiable as ALS Group USA, Corp., dba ALS Environmental. ALS Group USA Corp. is a component of ALS Limited, a publicly held Australian company. The ALS global website may be referred to for corporate ownership information (www.alsglobal.com/Our-Company). Organizational charts detailing the operational structure and reporting relationships in the laboratory are provided in Appendix B.

2.2 Avoiding Conflict of Interest through Organizational Structure

2.2.1 Through application of the policies and procedure outlined in this QA Manual and use of a defined organizational structure, the laboratory assures that it is impartial and that personnel are free from undue commercial, financial, or other undue pressures that might influence their technical judgment.

2.2.2 Policies are in place to prevent outside pressures or involvement in activities that may affect competence, impartiality, judgment, operational integrity, or the quality of the work performed at the laboratory.

2.2.3 Management and technical personnel have the authority and resources to carry out their duties and have procedures to identify and correct departures from the laboratory's management system.

2.2.4 Personnel understand the relevance and importance of their duties as related to the maintenance of the laboratory's management system. Ethics and data integrity procedure ensure that personnel do not engage in activities that diminish confidence in the laboratory's capabilities. Procedures and policies are also established to ensure confidentiality is maintained.

3) Management

The purpose of the QA program at ALS Environmental is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality.

3.1 Quality Policy Statement

The policy at ALS is to use good professional practices, to maintain quality, to uphold the highest standard of service, and to operate in accordance with these requirements and those of regulatory agencies, accrediting authorities, and certifying organizations. We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory. Laboratory management is committed to ensuring the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set forth in the *SOP for Laboratory Ethics and Data Integrity* (CE-GEN001) and in this Quality Assurance Manual (QAM). ALS Environmental is committed to operate in accordance with these requirements and those of regulatory agencies, accrediting authorities, and certifying organizations. The laboratory also strives for improvement through varying continuous improvement initiatives and projects.

Quality Management Systems are established, implemented and maintained by management. Policies and procedures are established in order to meet requirements of accreditation bodies and applicable programs as well as client's quality objectives. The laboratory's management is committed to complying with the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards (2003 and 2009 NELAP/TNI standards), ISO/IEC 17025:2005, and the Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory is involved. All personnel involved with environmental testing and calibration activities must familiarize themselves with the quality documentation and implement the policies and procedures in their work.

3.2 Quality Management Systems

The laboratory has developed a Quality Management System to ensure all products and services meet our client's needs. The system is implemented and maintained by the Quality Assurance Manager (QA Manager) with corporate oversight by the Corporate Quality Assurance Manager (CQAM). These systems are based upon ISO 17025:2005 standards, upon which fundamental programs (AIHA, TNI/NELAP, and DoD QSM) are based. Implementation and documentation against these standards are communicated in corporate policy statements, this QAM, and SOPs. Actual procedures, actions and documentation are defined in both administrative and technical SOPs. Figure 3-1 shows the relationships of the quality systems and associated documentation. Quality systems include:

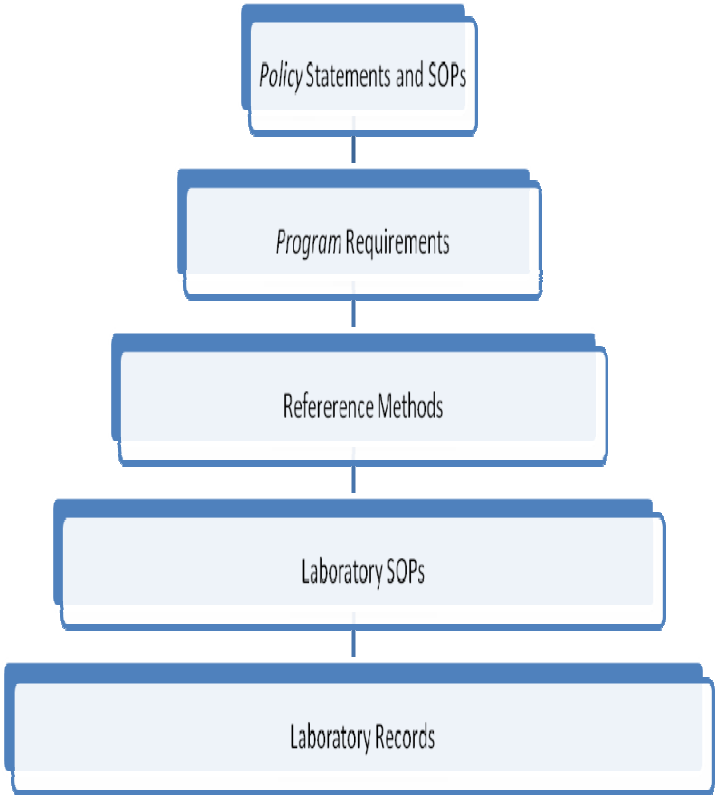
- Standard Operating Procedures
- Sample Management and Chain of Custody procedures
- Statistical Control Charting
- Standards Traceability
- Ethics Training
- Document Control
- Corrective Action Program
- Management Reviews
- Demonstration of Capability

The effectiveness of the quality system is assessed in several ways, including:

- Internal and External Audits covering all aspects of the organization
- Annual Management Reviews
- Analysis of Customer Feedback
- Internal and External Proficiency Testing

Figure 3-1

Relationships of Quality Management Systems and Documentation



3.3 Technical Elements of the Quality Assurance Program

The laboratory's technical procedures are based upon procedures published by various agencies or organizations (See Section 23). The Quality Assurance Program provides laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained by the QA department. This includes method approvals and audit

administration. In addition, internal audits are performed to assess compliance with policies and procedures. SOPs are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix I and SOPs in Appendix G.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, technical training and preventive maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

3.4 Professional Conduct

One of the most important aspects of the success of ALS Environmental is the emphasis placed on the integrity of the data provided and the services rendered. This success is reliant on both the professional conduct of all employees within ALS Environmental as well as established laboratory practices.

To promote quality, ALS Environmental requires certain standards of conduct and ethical performance among employees. The following examples of documented ALS Environmental policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action.
- Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible.
- It is the responsibility of all ALS Environmental employees to safeguard sensitive company information, client data, records, and information; and matters of national security concern should they arise. The nature of our business and the well-being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action. All employees sign a confidentiality agreement upon hire to protect the company and client's confidentiality and proprietary rights.

3.5 Prevention and Detection of Improper, Unethical or Illegal Actions

It is the intention of ALS Environmental to proactively prevent and/or detect any improper, unethical or illegal action conducted within the laboratory.

This is performed by the implementation of a program designed for not only the detection but also prevention. Prevention consists of educating all laboratory personnel of their roles and duties as employees, company policies, inappropriate practices, and their corresponding implications as described here.

In addition to education, appropriate and inappropriate practices are included in SOPs such as manual integration, data review and specific method procedures. Electronic and hardcopy data audits are performed regularly, including periodic audits of chromatographic electronic data. Requirements are described in the *SOP for Internal Audits* (CE-QA001) and details are listed in laboratory administrative SOPs. All aspects of this program are documented and retained on file according to the company policy on record retention.

The *SOP for Laboratory Ethics and Data Integrity* (CE-GEN001) also contains information on the ALS Environmental ethics and data integrity program, including mechanisms for reporting and seeking advice on ethical decisions.

3.6 Laboratory Data Integrity and Ethics Training

New employees are given a QA and Ethics orientation within the first month of hire. On an ongoing basis, all employees receive annual ethics refresher training. Topics covered are documented in writing and all training is documented. It is the responsibility of the QA Manager to ensure that the training is conducted as described.

Key topics covered are the organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues and record keeping. Training includes discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation.

Data integrity training provides assurance that a highly ethical approach to testing is a key component of all laboratory planning, method implementation, and training. There are four elements to the laboratory's procedures for data integrity. These include:

1) Data integrity training (conducted initially and at least annually);
2) Signed data integrity documentation for all employees;
3) In-depth periodic monitoring of data integrity;
4) Data integrity procedure documentation (*SOP for Laboratory Ethics and Data Integrity* (CE-GEN001)).

There is specific emphasis on the importance of proper written narration on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially deficient. A signature attestation sheet of data integrity training including their understanding of their obligations related to data integrity and as specified in the training is generated for attendees and maintained on file for review. Trainees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, or civil/criminal prosecution.

The training session includes many concepts and topics, numerous examples of improper actions (defined by DoD as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional), legal and liability implications (company and personal), causes, prevention, awareness, and reporting mechanisms.

3.7 Management and Employee Commitment

ALS Environmental makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the *SOP for Laboratory Ethics and Data Integrity* (CE-GEN001). This includes:

- ALS Environmental Open Door Policy – Employees are encouraged to bring any work related problems or concerns to the attention of local management or their Human Resources representative. However, depending on the extent or sensitivity of the concern, employees are encouraged to directly contact any member of upper management.
- FAIRCALL – An anonymous and confidential reporting system available to all employees that is used to communicate misconduct and other concerns. The program shall help minimize negative morale, promote a positive work place, and encourage reporting suspected misconduct without retribution. Associated upper management is notified and the investigations are documented.
- Use of flexible work hours. Within reason and as approved by supervisors, employees are allowed flexible work hours in order to help ease schedule pressures which could impact decision-making and work quality.
- Operational and project scheduling assessments are continually made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads. Procedures for subcontracting work are established, and within the ALS Environmental laboratory network additional capacity is typically available for subcontracting, if necessary.
- Gifts and Favors (Code of Conduct Agreement) – To avoid possible conflict of interest implications, employees do not receive unusual gifts or favors to, nor accept such gifts or favors from, persons outside the Company who are, or may be, in any way concerned with the projects on which the Company is professionally engaged.

All employees are required to sign and adhere to the requirements set forth in the *Code of Conduct Agreement, Confidentiality Agreement,* and *Ethics and Data Integrity Agreement*. The *Ethics and Data Integrity Agreement* is signed by all employees on an annual basis (see Appendix C).

3.8 The ALS Environmental-Simi Valley staff, consisting of approximately 30 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds, experience, and provide the comprehensive skills that the laboratory requires. As seasonal workload increases, temporary employees may be hired to perform specific tasks.

ALS Environmental is committed to providing an environment that encourages excellence. All employees share the responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 3-1 lists the ALS Environmental-Simi Valley personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program including ensuring compliance with ISO/IEC 17025:2005 and is responsible for overall laboratory efficiency and the financial performance of the Simi Valley facility.

  The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

- The **Quality Assurance Manager** (QA Manager) has the authority and responsibility for implementing, maintaining, and improving the quality system. This includes coordination of QA activities within the laboratory, ensuring that all personnel understand their contributions to the quality system, ensuring communication takes place at all levels within the laboratory regarding the effectiveness of the quality system, evaluating the effectiveness of training; and monitor trends and continually improve the quality system. Audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews can all be used to support quality system implementation. The QA Manager is responsible for ensuring compliance with all applicable regulatory compliance quality standards (i.e. NELAP/TNI, ISO/IEC 17025:2005, DoD QSM, etc.). The QA Manager works with laboratory staff to establish effective quality control and assessment plans and has the authority to stop work in response to quality problems. The QA Manager is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and ensuring the annual review of technical SOPs; maintaining QA records such as metrological records, archived logbooks, PT results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; and performing internal QA audits.

  The QA Manager reports directly to the Laboratory Director and also reports indirectly to the Manager of Quality Assurance, USA. It is important to note that when evaluating data, the QA Manager does so in an objective manner and free of outside, or managerial, influence.

- The Manager of Quality Assurance, USA is responsible for the overall QA program at all the ALS Environmental Group laboratories. The Manager of Quality Assurance, USA is responsible for oversight of QA Managers regulatory compliance efforts (NELAP/TNI, ISO, DoD, etc) and may perform internal audits to evaluate compliance. The Manager of Quality Assurance, USA approves company-wide SOPs and provides assistance to the laboratory QA staff and laboratory managers as necessary.

➢ In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are a Project Manager or Volatile Organics Technical Manager (for the Laboratory Director) and the Laboratory Director (for the QA Manager).

- In the event that work is stopped in response to quality problems, only the Laboratory Director or QA Manager have the authority to resume work.

- The **Environmental Health and Safety Coordinator** (EH&S) is responsible for the administration of the laboratory health and safety policies.

  This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S Coordinator is also designated as the Chemical Hygiene Officer. The EH&S Coordinator has a dotted-line reporting responsibility to ALS North America EH&S Director.

- The **Data Validation Coordinator/Reporting Supervisor** is responsible for data review, data package preparation, review and coordination, and preparation of case narratives (based on the information provided by the laboratory).

- The **Client Services Manager** is responsible for the Client Services Department defined for the laboratory (i.e. Project Managers, data reporting, etc.) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specifications to final deliverables. Sample management handles all activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.

- The **Project Manager** is a scientist assigned to each client to act as a technical liaison between the client and the laboratory. The Project Manager is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the ALS Environmental laboratory and administrative staff to ensure that client-specific needs are understood and that the services ALS Environmental provides are properly executed and satisfy the requirements of the client.

- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a QC program meeting department needs. Each **Department Manager and Supervisor** has the responsibility to ensure compliance with ISO/IEC 17025:2005, ensure that QC functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.

- The **Sample Management Office** plays a key role in the laboratory QA program by performing and/or assisting in the proper preparation and shipment of sampling media. In addition, personnel are responsible for the verification of sample receipt information, performing sample acceptance and log-in and distribution of documentation per laboratory defined procedures and the initial storage of samples in the proper environment and location and performing proper sample

disposal. Responsibilities also include monitoring and recording of critical thermal preservation equipment temperatures and calibration of associated thermometers against NIST traceable thermometers.

- **Information Technology** (IT) staff is responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.
- The **Procurement Manager** is responsible for directing and coordinating activities of personnel engaged in buying materials and supplies.

Table 3-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Kelly Horiuchi, B.A.	15	Laboratory Director / Project Manager
Chaney Humphrey, B.S.	11	Quality Assurance Manager
Robin Gill	35	Data Validation Coordinator / Reporting Supervisor
Ku-Jih Chen, B.S.	40	Principle Chemist
Sue Anderson, B.S.	25	General (WET) Chemistry Technical Manager / Project Manager
Samantha Henningsen, B.S.	6	Project Manager
Kathleen Aguilera, B.A.	26	Client Services Manager / Project Manager
Wade Henton, B.S.	29	Volatiles (GC) Technical Manager
Chris Parnell, B.S.	29	Operations Manager / Volatiles (GC/MS) Technical Manager
Wida Ang, B.S.,M.S.	30	Volatiles (GC/MS) Team Leader
Madeleine Dangazyan, B.S.	20	Semi-Volatiles / Industrial Hygiene Technical Manager
Jeff Christian, B.S.	36	Director of Operations – Western U.S.

Additional Key Personnel	Project Role
Joe Caulfield	LIMS Manager
Steve Manak	Procurement Group Leader

4) Document Control

4.1 Procedures for control and maintenance of documents are described in the *SOP for Document Control* (CE-GEN005). The requirements of the SOP apply to all laboratory logbooks (standards, maintenance, run logbooks, etc), certificates of analysis, SOPs, QAMs, quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled ALS Environmental documents.

4.2 The contents of this manual are reviewed, revised (as needed) and approved for use at least annually by authorized personnel (QA Manager, Laboratory Director, and Technical Directors) where the scope of the review ensures that it continuously reflects current policies and practices and incorporates all applicable requirements. Additionally, the date the review was completed is indicated by the date of the last approval signature on the title page.

4.3 Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the QA Manager, or designee, and ensures that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file. Logbook entries are standardized following the *SOP for Making Entries onto Analytical Records* (CE-QA007). The entries made into laboratory logbooks are reviewed and approved at a regular interval (quarterly).

4.4 A records system is used which ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data and Record Archiving* (ADM-ARC).

4.5 External documents relative to the management system are managed by the QA Manager. To prevent the use of invalid and/or outdated external documents, the laboratory maintains a master list of current documents and their availability. The list is reviewed before making the documents available. External documents are not issued to personnel.

4.6 Electronic Signatures It is a policy of ALS Environmental to allow the use of electronic signatures. For data reporting an electronic signature may be applied to the report by an approved report signatory and is binding to the same extent as a handwritten wet signature.

To authenticate the electronic signature the identity of the signatory is verified before their electronic signature can be created. Each electronic signature shall be unique to a single individual and shall not be used by any other individual. These signatures are established using only defined procedures within the software and are verified using the two distinct components of *username* and *password*. Each use of the electronic

signature requires entry of the username and the password. The report may not be changed once the signature has been applied.

Additionally, as a form of 'signature' used for LIMS, email, and certain internal documentation processes (e.g. acknowledgements, attestations, audit trails, etc.), and other electronic tools the user's system login credentials are used to verify and authenticate the identity of the user. Following login, these credentials are used to identify and document the user.

5) Review of Requests, Tenders and Contracts

5.1 Procedure for the Review of Work Requests

5.1.1 Requests for new work are reviewed prior to signing any contracts or otherwise agreeing to perform the work. The specific methods to be used are agreed upon between the laboratory and the client. A capability review is performed to determine if the laboratory has or needs to obtain certification to perform the work, to determine if the laboratory has the resources (personnel, equipment, materials, capacity, skills, expertise) to perform the work, and if the laboratory is able to meet the client's required reporting and QC limits. The results of this review are communicated to the client and any potential conflict, deficiency, lack of appropriate accreditation status, or concerns of the ability to complete the client's work are resolved.

5.1.2 Any differences between the request or tender and the contract shall be resolved before any work commences. The client should be notified at this time if work is expected to be subcontracted. Each contract shall be acceptable both to the laboratory and the client. Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work.

5.1.3 If a contract needs to be amended after work has commenced, the contract review process is repeated and any amendments are communicated to all affected personnel. Changes in accreditation status affecting ongoing projects must be reported to the client.

5.2 Allowed Deviations from Standard Operating Procedures

5.2.1 When a client requests a modification to an SOP the Project Manager must discuss the proposed deviation with the laboratory supervisor and obtain approval to accept the project. The Laboratory Director and QA Manager may also be involved. The Project Manager is responsible for documenting the approved or allowed deviation from the SOP.

5.2.2 When a client request necessitates a deviation or departure from company policies or procedure involving any non-technical function, the allowed deviation must be approved by the laboratory or the Laboratory Director. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the Laboratory Director will address the possible need for a change in policy.

6) Subcontracting of Tests

Analytical services are subcontracted when the laboratory needs to balance workload or when the requested analyses are not performed by the laboratory. Subcontracting, to capable qualified laboratories is only done with the knowledge and approval of the client.

Subcontracting to another ALS Environmental laboratory is preferred over external-laboratory subcontracting. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories* (CE-QA004). The QA Manager is responsible for maintaining a list of qualified subcontract laboratories.

7) Purchasing Services and Supplies

The quality level of reagents and materials (grade, traceability, etc.) required is specified in the analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. The *SOP for Handling Consumable Materials* (ADM-CONSUM) provides default expiration requirements. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in the *SOP for Procurement and Control of Laboratory Services and Supplies* (CE-GEN007). Also, refer to section 13.5 for a discussion of reference materials.

Receipt procedures include technical review of the purchase order/request to verify that what was received is identical to the item ordered. The laboratory checks new lots of reagents for unacceptable levels of contamination prior to use in sample preservation, sample preparation, and sample analysis by following the *SOP for Quality of Reagents and Standards* (CE-QA012).

8) Service to the Client

The laboratory uses a number of systems to assess its daily operations. In addition to the routine quality control (QC) measurements, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients including; on-time performance, customer complaints, training reports and non-conformity reports. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

ALS Environmental utilizes a number of different methods to ensure that adequate resources are available for service demands. Senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating sufficient resources. All Requests for Proposal (RFP) documents are reviewed by Project Managers, Business Development and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Project Plans (QAPPs) if available. Status/production meetings are also conducted regularly with the laboratory and project managers to inform the staff of the status of incoming work, future projects, or project requirements.

When a customer requests a modification to an SOP, policy, or standard specification the Project Manager will discuss the proposed deviation with the Laboratory Director and department manager to obtain approval for the deviation. The QA Manager may also be involved. All project-specific requirements must be on-file and with the service request upon logging in the samples. The modification or deviation must be documented. A Project-Specific Communication Form, LIMS comments, or similar, may be used to document such deviations.

The laboratory shall afford clients cooperation to clarify the client's request and to monitor the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients. The laboratory maintains and documents timely communication with the client for the purposes of seeking feedback and clarifying customer requests. Feedback is used and analyzed to improve the quality of services. The *SOP for Handling Customer Feedback* (CE-GEN010) is in place for these events.

9) Complaints

The laboratory maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the Project Manager) is responsible for documenting the complaint. If the Project Manager is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback* (CE-GEN010).

10) Facilities and Equipment

ALS Environmental-Simi Valley maintains approximately 20,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system is designed to meet any needs of analyses performed in the separate work areas. ALS Environmental-Simi Valley minimizes laboratory contamination sources by employing janitorial staff to ensure good housekeeping. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Sample Management Office; Shipping and Receiving
- Records Archival
- Volatile Organics Laboratory (GC and GC/MS)
- Semi-Volatiles Laboratory (GC, GC/MS and HPLC)
- Ultra-Low Level Volatile Organics GC/MS
- General/Wet Chemistry Laboratory
- R&D Laboratory
- Canister Conditioning and Maintenance
- Flow Controller and Critical Orifice Calibration Station
- Sample Storage Walk-in Refrigerator
- Sample, Standards, and Media Storage
- Waste Disposal
- Laboratory Deionized Water System
- Laboratory Management, Client Service, Report Generation and Administration
- Information Technology (IT)

The designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Refer to Appendix D for facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix E lists the major equipment, illustrating the laboratory's overall capabilities and depth.

ALS Environmental-Simi Valley also maintains a satellite extraction facility located at 2360 Shasta Way, Unit G, Simi Valley, California. The approximately 2,000 square foot building contains five fume hoods and is designed with the purpose of performing semi-volatile

organics extraction of air, liquid and solid matrices. The extraction facility is equipped with sufficient bench space, glassware washing equipment and materials, flammable solvent storage, sample/extract storage refrigerators and an electric kiln. Refer to Appendix D for the floor plan of the facility.

10.1 Preventive Maintenance

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at ALS Environmental (e.g., GC/MS systems, gas and liquid chromatographs, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at ALS Environmental contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at ALS Environmental before it may be used for sample analysis. Each instrument must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument or if the continuing calibration verification acceptance criteria have not been met as specified in the standard operating procedure. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at ALS Environmental. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the department supervisor or laboratory director. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the department supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. The laboratory maintains an adequate supply of expendable maintenance items (expected lifetime of part of less than 1 year.) These parts include items needed to perform the preventive maintenance procedures listed in Table 16-1.

When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and

- Demonstration of return to analytical control.

See the Table 16-1 for a list of preventive maintenance activities and frequency for each instrument.

For further information regarding Instrumentation see the *SOP for Analytical Instrument Acquisition, Reassignment, Maintenance and Documentation* (ADM-INSTRUM).

10.2 Temperature Control

Temperatures are monitored and recorded for all critical measurement temperature-regulating devices including freezers, refrigerators and ovens. Each piece of equipment is labeled with a unique identifier, the required temperature or range of use according to the needs of the analysis or application. Temperature record books are kept which contain equipment identifier, daily-recorded temperatures (if in use, business days), acceptance criteria and the initials of the laboratory staff member who performed the checks for all temperature-regulating devices in daily use.

10.3 Water Purification Systems

Purified water is utilized for a number of laboratory functions including instrument and method blanks, trip blanks, washes and sample dilutions. The water purification system utilizes three mixed-ion beds, four filters, and resistively lights with constant water recirculation. It is designed to produce deionized water of ASTM Type II quality, with 16-18 megohm-cm resistance at 25˚C and is checked and recorded daily (prior to and if in use). Maintenance and repair on the system is conducted by an approved service supplier and all records including purification checks/verifications are maintained on file for review. For procedures on additional purification (i.e., boiling and/or purging) and purification checks/verifications, refer to the applicable method standard operating procedures.

11) Sample Management

Standard operating procedures have been established for all aspects of sample management within the laboratory including sample receiving, handling, acceptance, log-in, protection, storage, retention, transportation, and disposal. The procedures include provisions necessary to protect the integrity of the sample (as received) and to protect the interests of the laboratory as well as the client. These procedures ensure that samples are handled properly and that all associated documentation is complete and consistent. The sample handling factors that must be taken into account to ensure accurate, defensible analytical results include but are not limited to:

- Amount of sample taken (sampling)
- Type of container used
- Existence and type of sample preservation
- Holding Time
- Proper custodial documentation
- Sample storage, tracking and/or transfer
- Retention
- Disposal

A record of all procedures to which a sample is subjected while in the possession of the laboratory including acceptance, rejection, login, identification, preservation checks, storage, tracking, and disposal are documented and maintained. In addition, all indirect procedures which support each record of a sample and protects the integrity of a sample is documented and maintained (i.e., refrigerator and freezer temperature checks, thermometer calibrations, etc.).

11.1 Sampling

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples.

ALS Environmental-Simi Valley does not provide sampling services. The laboratory only provides materials needed for sample collection; therefore, ALS Environmental-Simi Valley recommends that clients follow sampling guidelines described in the specific reference methods including 40 CFR 136 and/or USEPA SW-846, NIOSH, OSHA, ASTM, CARB and SCAQMD as appropriate.

When transporting samples to the laboratory, the most expedient but lawful route of transport should be utilized. Also, the hazardous potential of the samples needs to be considered when shipping samples via air freight or passenger airlines.

11.2 Preservation

ALS Environmental-Simi Valley uses sample preservation, container, and holding time recommendations published in a number of referenced documents including, but not limited to USEPA SW 846, USEPA 600/4-79-020, USEPA 600/r-93-100 (inorganic substances), 600/4-91-010, and EPA/625/R-96/010b (air samples) and the US EPA Methods Update Rule effective 4/11/07. The complete citation for each of these and other references can be found in Section 23 of this document. The appropriate container, preservation and holding time information are summarized in Appendix F. Additional information on this is addressed in each corresponding method SOP.

11.3 Shipping of Containers and Samples

ALS Environmental-Simi Valley provides sample containers to clients via media requests for all matrices (soil, water, air) with the appropriate preservatives (as applicable). These containers include Tedlar bags, Summa canisters, silica-gel tubes, etc. ALS Environmental-Simi Valley keeps client-specific shipping requirements on file and utilizes all major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. ALS Environmental-Simi Valley also provides its own courier service that makes scheduled courier runs in the greater Los Angeles metropolitan area. The procedures for all requirements directed toward media requests follow the requirements detailed in the *SOP for Media Request Fulfillment* (ADM-Media_Req).

11.4 Sample Receiving and Acceptance

It is the policy of ALS Environmental-Simi Valley to check and record the condition of each sample (i.e. pressure, temperature, etc.) delivered to the Sample Management Office (SMO) and received by the Sample Management Custodian or alternates against certain acceptance criteria as documented in the *SOP for Sample Receiving, Acceptance, and Log-In* (SMO-SMPL_REC). This policy is available to all sample management personnel for reference. Any samples, which deviate from these outlined areas, will be clearly flagged with the nature and substance of the deviation. Assessment and condition checks utilized by ALS Environmental-Simi Valley for the acceptance or rejection of samples are based on the criteria found in Appendix F, applicable Quality Assurance Project Plan (QAPP), permit, program or rule where appropriate. This verification of sample integrity is conducted by the Sample Custodian and may be dependent on the matrix (i.e., temperature, preservation, and headspace) being submitted.

Any abnormalities or departures from specified condition requirements (as described herein) as observed during the initial assessment are recorded. When there is any doubt as to the suitability of a sample for testing, including signs of damage, when a sample does not conform to the description provided, or when the test method required is not specified in sufficient detail the appropriate Project Manager (PM) is notified.

The Project Manager is to consult with the client, whenever possible, regarding specific integrity issues documented during sample receipt for further instructions before proceeding and retain a written record of discussion. There may be instances where the client is unavailable, in which case the PM shall document all attempts at contacting the client.

There may be a need to inform the client that a sample(s) is rejected and cannot be accepted for analysis into the laboratory. This situation includes, but is not limited to loss of sample or insufficient amount (subsampling may be performed if it would not cause loss of sample integrity, but the procedure must be indicated with the test results). Subsampling as in the case of air samples is not appropriate.

The procedures for sample documentation, handling acceptance requirements and deviations from the sample acceptance policy are discussed in detail in the *SOP for Sample Receiving, Acceptance and Log-In* (SMO-SMPL_REC). This procedure is also in place to ensure samples are received and properly logged into the laboratory, and that all associated sample documentation, including Chain-of-Custody (COC) records are complete and consistent with the samples received. All associated documentation, including chain of custody forms, memos, transmittal forms, and phone logs, are kept with each project file.

11.5 Sample Log-in

Each sample is logged into the laboratory in such a way as to ensure traceability and cross-reference with regards to the unique laboratory job number, sample identifications and client sample identifications. The laboratory identification is retained throughout the life of the sample in the laboratory. The identification system is designed and operated to ensure that samples cannot be confused physically or in laboratory documentation. Additional information is provided in the *SOP for Sample Receiving, Acceptance, and Log-In* (SMO_SMPL_REC).

11.6 Sample Custody

A sample is in someone's "custody" if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked up so that no one can tamper with it;
4. It is kept in a secured area, restricted to authorized personnel only.

Chain-of-Custody (COC) records are used to establish the legal custody of samples, showing the continuous possession of samples from sample collection and transportation to final destination at the laboratory. Custody of each sample is maintained from receipt through disposal (internally utilizing LIMS). When environmental samples are shipped to other laboratories for analysis, the sample management office follows formalized procedures for maintaining the chain of custody, which is written in SOPs for Sample Receiving, Acceptance and Login and Laboratory Storage, Analysis, and Tracking.

When samples are removed from the fixed lab and transported to the off-site extraction facility for sample preparation, internal chain of custody procedures still apply. When sample preparation is completed, sample extracts are returned to the laboratory.

Laboratory security and access is important in maintaining the integrity of samples received at ALS Environmental-Simi Valley.

Access to the building is limited to the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area.

The laboratory is equipped with an alarm system which is monitored by a private security firm who provides nighttime and weekend security.

11.7 Sample Storage, Analysis and Tracking

The procedures and requirements for documenting the storage, analysis and tracking as well as maintaining integrity of samples are detailed in the *SOP for Laboratory Storage, Analysis, and Tracking* (ADM-LabSAT).

11.8 Sample Retention and Waste Disposal

Upon completion of all analyses, the laboratory samples are retained in accordance with the requirements specified in the method SOPs and the *SOP for Waste Disposal* (ADM-Waste). The samples are disposed according to approved disposal practices or returned to the client (if applicable). All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. This evaluation is generally based on results from analyses performed on the sample by ALS Environmental-Simi Valley or an approved subcontract laboratory. It should be noted that all wastes produced at the laboratory, including the laboratory's own various hazardous waste streams, are treated in accordance with all applicable local, State and Federal laws. Complete documentation is maintained for samples from initial receipt through final disposal. This ensures an accurate record of the samples from "cradle to grave."

11.9 Intra-laboratory / Inter-laboratory Transfer of Samples

When environmental samples are shipped to another laboratory for analysis, samples are properly packed for shipment and preserved if necessary. Sample bottles are wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during the transportation process. Blue or wet ice is used for temperature preservation, where necessary.

Figure 11-1
Air Chain of Custody Form

Air - Chain of Custody Record & Analytical Service Request

Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

Company Name & Address (Reporting Information)

Project Name

Project Number

ALS Contact:

Analysis Method

Project Manager

P.O. # / Billing Information

Phone | Fax

Email Address for Result Reporting

Sampler (Print & Sign)

Comments
e.g. Actual Preservative or specific instructions

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Canister ID (Bar code # - AC, SC, etc.)	Flow Controller ID (Bar code #- FC #)	Canister Start Pressure "Hg	Canister End Pressure "Hg/psig	Sample Volume			

Report Tier Levels - please select

Tier I - Results (Default if not specified) _____ Tier III (Results + QC & Calibration Summaries) _____
Tier II (Results + QC Summaries) _____ Tier IV (Data Validation Package) 10% Surcharge _____

EDD required Yes / No
Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:	
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:	Cooler / Blank Temperature ____°C

Figure 11-2
Soil / Water Chain of Custody Form

Soil / Water - Chain of Custody Record & Analytical Service Request Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

ALS Contact:

Company Name & Address (Reporting Information)

Project Name

Project Number

Project Manager

P.O. # / Credit Card / Billing Information

Phone | Fax

Email Address for Result Reporting

Analysis

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Water	Soil	Solid	Other				Comments

Report Tier Levels - please select
Tier I - Results (Default if not specified) ____ Tier III (Results + QC & Calibration Summaries) ____ EDD required Yes / No
Tier II (Results + QC Summaries) ____ Tier IV (Data Validation Package) 10% Surcharge ____ Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs)

Reliquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Reliquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Reliquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:

Cooler / Blank / Ice / No Ice
Temperature _____ °C

Figure 11-3

ALS Environmental
Sample Acceptance Check Form

Client: _____ Work order: _____
Project: _____
Sample(s) received on: _____ Date opened: _____ by: _____

Note: This form is used for all samples received by ALS. The use of this form for custody seals is strictly meant to indicate presence/absence and not as an indication of compliance or nonconformity. Thermal preservation and pH will only be evaluated either at the request of the client and/or as required by the method/SOP.

		Yes	No	N/A
1	Were **sample containers** properly marked with client sample ID?	☐	☐	☐
2	Container(s) **supplied by ALS**?	☐	☐	☐
3	Did **sample containers** arrive in good condition?	☐	☐	☐
4	Were **chain-of-custody** papers used and filled out?	☐	☐	☐
5	Did **sample container labels** and/or tags agree with custody papers?	☐	☐	☐
6	Was **sample volume** received adequate for analysis?	☐	☐	☐
7	Are samples within specified holding times?	☐	☐	☐
8	Was proper **temperature** (thermal preservation) of cooler at receipt adhered to?	☐	☐	☐
9	Was a **trip blank** received?	☐	☐	☐
10	Were **custody seals** on outside of cooler/Box?	☐	☐	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☐
	Were signature and date included?	☐	☐	☐
	Were seals intact?	☐	☐	☐
	Were custody seals on outside of sample container?	☐	☐	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☐
	Were signature and date included?	☐	☐	☐
	Were seals intact?	☐	☐	☐
11	Do containers have appropriate **preservation**, according to method/SOP or Client specified information?	☐	☐	☐
	Is there a client indication that the submitted samples are **pH** preserved?	☐	☐	☐
	Were **VOA vials** checked for presence/absence of air bubbles?	☐	☐	☐
	Does the client/method/SOP require that the analyst check the sample pH and if necessary alter it?	☐	☐	☐
12	**Tubes:** Are the tubes capped and intact?	☐	☐	☐
	Do they contain moisture?	☐	☐	☐
13	**Badges:** Are the badges properly capped and intact?	☐	☐	☐
	Are dual bed badges separated and individually capped and intact?	☐	☐	☐

Lab Sample ID	Container Description	Required pH *	Received pH	Adjusted pH	VOA Headspace (Presence/Absence)	Receipt / Preservation Comments

Explain any discrepancies: (include lab sample ID numbers): _____

RSK - MEEPP, HCL (pH<2); RSK - CO2, (pH 5-8); Sulfur (pH>4)

12) Analytical Procedures

ALS Environmental employs methods and analytical procedures from a variety of external sources. Reference documents include but are not limited to: ASTM, CARB, NCASI, NIOSH, OSHA, SCAQMD, USEPA SW-846, USEPA 600/4-79-020, 600/4-91-010, 600/R-93/100 (inorganic substances), 600/625/R-96/010b (air samples), EPA 40 CFR part 136 and associated Method Update Rules and Supplements, and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 23. Other published procedures, such as state-specific methods, program-specific methods, or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by ALS Environmental is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix J. Further details are described below.

12.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks

ALS Environmental maintains SOPs for use in both technical and administrative functions (Refer to Appendix G). SOPs are written following standardized format and content requirements as described in the *SOP for Establishing Standard Operating Procedures* (CE-GEN009). Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the QA Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents, unless otherwise noted. The procedures for document control are described in the *SOP for Document Control* (CE-GEN005). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries onto Analytical Records* (CE-QA007). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

12.2 Modified Procedures

ALS Environmental strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

12.3 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that ALS Environmental-Simi Valley has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.
2) All (field) samples in a batch are of the same matrix.
3) The QC samples to be processed with the (field) samples include:
   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination

   b) Laboratory Control Sample

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.
4) A single lot of reagents is used to process the batch of samples.
5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.
6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours between the start of processing of the first and last sample of the batch.
7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.
8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).
9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.
10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.
11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

Note: Matrix spiked samples are often not feasible for air matrices. Therefore, the MS shall be used as required by the test method and as specified by the corresponding method SOP.

12.4 Specialized Procedures

ALS Environmental not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are specialized GC/MS analyses, and low level organics analyses (including PAHs, pesticides and PCBs).

12.5 Demonstration of Capability

A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria are met.

12.6 Method Detection Limits and Method Reporting Limits & Limits of Detection/ Quantitation

Method Detection Limits (MDL) for methods performed at ALS Environmental-Simi Valley are determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation* (CE-QA011), which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. limit of quantitation - LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

13) Measurement Traceability and Calibration

All equipment and instruments used at ALS Environmental are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files. Records are maintained to provide traceability of reference materials.

Traceability is defined as the property of a measurement result or value of a standard which can be related to stated references through an unbroken chain, each with stated uncertainties and is documented for all material used to perform calibrations. The documentation, a certificate of analysis containing, at a minimum, the manufacturer, address, accreditation number (where applicable), how traceability was achieved, the traceable values, their associated uncertainty, and the unique serial or laboratory identification number of the equipment or standard reference material (SRM) shall serve as initial point in the chain of traceability. The unique serial number or laboratory identification number is used throughout the laboratory to trace equipment and materials back to the original certificate of analysis.

Laboratory support equipment (thermometers, balances, and weights) are verified on an annual basis by a vendor accredited to ISO/IEC 17025:2005 International Standards. All analytical measurements generated at ALS Environmental are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

Equipment subjected to overloading or mishandling, or has been shown by verification to be defective, is taken out of service and labeled until repaired. That piece of equipment is placed back in service only after verifying, by calibration, that it performs satisfactorily.

13.1 Temperature Measuring Devices

All thermometers are identified by a unique identifying number (i.e., serial number), and the calibration of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. All corresponding correction factors are noted on the device as well as in the thermometer calibration logbook. The NIST calibrated thermometer is recertified by an approved vendor accredited ISO/IEC 17025:2005 International Standard on an annual basis and certificates are retained on file for review. All temperature monitoring is conducted in accordance with the *SOP for Sample Receipt, Acceptance and Log-In* (SMO-SMPL_REC) and thermometer calibration requirements are performed in accordance with the *SOP for Calibration and Use of the Laboratory Support Equipment* (ADM-SupEQ).

A number of thermometers include a temperature range per certain project requirements (complies with Department of Defense Quality Systems Manual for Environmental Laboratories); this range is recorded to document consistent compliance with required temperatures for refrigerators and freezers, where applicable.

13.2 Volumetric Dispensing Devices

The accuracy of pipettes used to make critical-volume measurements is verified on a quarterly basis. Typically, the indicated volume or range (where applicable) of the pipette is checked and both the accuracy and precision verification are performed using the above-mentioned procedure. The calibrations are evaluated against the intended use (volume or range) of the pipette and if the calibration is not approved for the specified volume(s) it is tagged accordingly (i.e. "Do Not Use Below 5uL"). The results for all calibration verifications are recorded and maintained.

Note: Glass microliter syringes including gas-tight syringes are considered in the same manner as Class A glassware and are not held to the calibration/verification requirements as are other volumetric dispensing devices.

13.3 Analytical Balances and Weights

Analytical balances and weights are calibrated/recertified and certificates issued annually by an approved vendor accredited to ISO/IEC 17025:2005 International Standard. The calibration of each balance is checked once each day of use in the expected range, utilizing the calibrated weights. Bound record books are kept which contain the identification of balance (serial number), recorded measurements and the initials of the analyst who performed the check. All certificates for the balances and weights are available for review.

13.4 Pressure/Vacuum Gauges

ALS Environmental-Simi Valley digital pressure/vacuum gauges are used in a number of critical measurements within the laboratory. The following is a list of the uses for this gauge type.

- Canister cleaning and conditioning
- Measure the vacuum on canisters before they are sent to the client for sampling.
- Measure the initial/final vacuum/pressure of canisters prior to analysis.
- Measure pressure during the preparation of selected standards.

Digital pressure/vacuum gauges are calibrated and certificates issued once per year by an approved metrology organization. All calibrations are performed against standards traceable to the National Institute of Standards and Technology (NIST) or other recognized national metrology institutes. In addition, ALS Environmental-Simi Valley performs a calibration check for each gauge six months following the calibration date. The laboratory retains all corresponding calibration and verification documentation for review.

13.5 Source and Preparation of Standards and Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors where possible have fulfilled the requirements for ISO 9001 certification and/or are ISO 17025 accredited. ALS Environmental-Simi Valley relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination.

The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries onto Analytical Records* (CE-QA007). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material.

13.6 Instrument Calibration

The laboratory specifies the procedures and documentation for initial instrument calibration and continuing calibration verification in the applicable method standard operating procedures to ensure that data is of known quality and is appropriate for a specific regulation and/or client requirement. The procedural steps for calibration including, frequency, number of points, integration, calculations, acceptance criteria (appropriate to the calibration technique employed), corrective action, associated statistics, and data qualifications are included in applicable methods, method standard operating procedures and/or client project plans. The essential elements that define the procedures and required documentation for initial instrument calibrations are specified below.

- Sufficient raw data records are retained to permit reconstruction of all calibrations.
- If a reference or mandated method does not specify the number of calibration standards, the initial calibration range shall consist of a minimum of 5 contiguous calibration points for organics and a minimum of 3 contiguous calibration points for inorganics. The actual numbers of points utilized is specified in the corresponding method SOP.
- The concentrations should bracket the expected concentration range of samples.
- Initial instrument calibration procedures referenced in test methods (either directly or indirectly) are readily available to the analysts.
- All sample results are quantitated from the initial instrument calibration and are not quantitated from any continuing instrument calibration verification unless otherwise specified by regulation, method or program.
- The initial instrument calibration is verified with a standard obtained from a second manufacturer or lot and traceability to a national standard is maintained, where available.
- The acceptance criteria utilized is appropriate for the calibration technique employed.
- The lowest calibration standard in the initial calibration is at or below the lowest concentration for which quantitative data are to be reported and is referred to at this laboratory as the method reporting limit (MRL). Some programs and/or agencies refer to this limit as the practical quantitation limit (PQL) or Limit of Quantitation (LOQ).
- Any data reported below the MRL or above the highest calibration standard is considered to have an increased quantitative uncertainty and is appropriately qualified in the report.

- The lowest calibration standard is above the limit of detection or method detection limit (MDL).

13.7 Internal and External Calibrations

Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area of the target compound in the sample or sample extract to the peak area of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF) or relative response factor (RRF) in some methods.

External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector responses to the amount (mass) of analyte in the calibration standard is defined as the calibration factor or in some cases it may be referred to as response factor.

13.8 Continuing Calibration Verification

The essential elements that define the procedures and required documentation for continuing instrument calibration verification are specified below.

- When an initial calibration is not performed on the day of analysis, continuing instrument calibration verification is analyzed with each batch.
- Calibration is verified for each reported compound, element or parameter; however, for multi-component analytes such as aroclors or total petroleum hydrocarbons a representative chemical related substance or mixture may be used. The allowance for this exception is dependent on applicable regulatory, method, or client project plans.
- Generally, the instrument calibration verification is performed at the beginning, end, and every ten samples of each analytical batch (except, if an internal standard is used, only one verification needs to be performed at the beginning of the analytical batch); whenever it is suspected that the analytical system may be out of calibration; if the time period for calibration or most previous calibration verification has expired; or for analytical systems that contain a specific calibration verification requirement. Specific requirements for the frequency of continuing calibration verification, for a particular method, is specified in the corresponding method standard operating procedure.

14) Assuring the Quality of Results

A primary focus of ALS Environmental's QA Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. ALS Environmental has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

14.1 Quality Control Objectives

14.1.1 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination).

ALS Environmental utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement.

14.1.2 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

14.1.3 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the QA Manager. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

14.1.4 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. ALS Environmental has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample. Air samples received by the laboratory in canisters and bags are considered to be homogenous and therefore, no special sample preparation procedures are necessary.

14.1.5 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using ALS Environmental or project-specified data qualifiers.

14.2 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below.

14.2.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (air, water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

14.2.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

14.2.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed.

The frequency of CCB analysis is once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

14.2.4 Calibration Standards

Calibration standards are vapors, liquids or solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

14.2.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used for calibration. Once it is determined that there is no defect or error in the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

14.2.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

14.2.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

14.2.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

14.2.9 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free liquid, solid or air matrix to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

14.2.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water samples.

14.2.11 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

Recovery (%) = (S - A) x 100 ÷ T

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

Note: Matrix spiked samples are often not feasible for air matrices. Therefore, the MS shall be used as required by the test method and as specified by the corresponding method SOP.

14.2.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample.

Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$\textit{Relative Percent Difference (RPD)} = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

14.2.13 Control Charting

The generation of control charts is routinely performed at ALS Environmental. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Limits* (CE-QA009).

14.2.14 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at ALS Environmental undergoes a rigorous cleansing procedure prior to every usage. The *SOP for Glassware Cleaning* (ADM-GLASS) has been generated and outlines the various procedures used at ALS Environmental-Simi Valley; each procedure is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

14.2.15 Collection Efficiency

In the case of sampling trains (consisting of one or more multi-section sorbent tubes), which are received intact by the laboratory, the “front” and “back” sections shall be separated if required by the client. Each section shall be processed and analyzed separately and the analytical results reported accordingly.

14.2.16 Desorption Efficiency and Method Reporting Limits (Industrial Hygiene)

Desorption efficiency (DE) is the ability of an analytical method to recover the analyte from the collection media. Desorption efficiencies are determined initially and for each analyte to be reported. In addition, a DE study is performed each time there is a change in the test method, or with each new lot of media. Desorption efficiency shall be determined using sorbent media from the same lot number used for the field samples, if possible, and of the identical size and type. The DE values are used to correct the sample results (for all samples except passive samplers) before reporting.

Minimum reporting limits for each reportable analyte are determined initially by the analysis of spiked media, prepared at the desired reporting limit and carried through the entire analytical process. The reporting limit is verified or re-established annually (or if there is a change in methodology or instrumentation) and instrument performance is checked with each analytical batch through the analysis of an analytical standard prepared at the reporting limit.

14.2.17 Field and Trip Blanks

Field and trip blanks are analyzed when they are submitted to the laboratory for analysis. The actual field samples are flagged (when analytes are found in the blank) if and only if the laboratory is able to analyze the samples in the same analytical sequence as the corresponding field or trip blank. If this is not possible due to client submission restrictions then the results for the samples and blanks shall be reported independently with no flag. However, an explanation of this is included in the final report. This laboratory does not feel that Summa canisters are suitable for use as trip blanks. It is for this reason that the results for these types of containers are reported as separate samples and flagging is not considered appropriate.

14.3 Uncertainty

When requested by the client or relevant to the validity of reported results, the estimation of measurement uncertainty will be provided to a client or regulatory agency. How the uncertainty will be reported may be dictated by the client's reporting specifications. Procedures for determining and reporting uncertainty are given in the *SOP for Estimation of Uncertainty of Analytical Measurements* (CE-QA010).

15) Control of Non-Conforming Environmental Testing Work

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s) (See Appendix H). Failure to meet established analytical controls, such as the quality control objectives, prompts corrective action. Corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, department manager, and/or the QA Manager may examine and pursue alternative solutions. In addition, the appropriate Project Manager is notified in order to ascertain if the client needs to be notified.

16) Corrective Action, Preventive Action, and Improvement

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using a *Nonconformity and Corrective Action Report* form. The procedure and responsibilities for addressing nonconforming work is defined in the *SOP for Nonconformance and Corrective Action* (CE-QA008). Nonconformances are reported to the client using various means (voice, email, narrative, etc). When a nonconformance occurs that casts doubt on the validity of the test results or additional client instructions are needed, the Project Manager notifies the client the same business day that the nonconformance is confirmed and reported. The QA Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the QA Manager. The QA Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate Project Manager is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

Part of the corrective action process involves determining the root cause. Identifying the root cause of a nonconformance can be difficult, but important for implementing effective corrective action. Root cause principles are used to determine assignable causes, which leads to corrective action taken to prevent recurrence.

16.1 Preventive Action and Improvement

Various preventive action and improvement processes are used for eliminating potential problems or averting problems before they occur. This is explained in the *SOP for Preventive Action* (CE-GEN004).

Table 16-1

Equipment Maintenance Procedures

Instrument	Applicable Activity	Frequency	Performed
Gas Chromatographs	Replace septum	As required	In-House and Outside Vendor
	Check system for gas leaks, loose/fray wires and insulation	With cylinder change/Open system	
	Replace injection port liner	As required	
	ECD wipe test	Every 6 months	
	Thermally Clean ECD	As needed	
	Clean FID	As required	
	Change TCD assembly	As required	
	SCD – Change reaction tube	As required	
	Catalyst check	As required	
Gas Chromatography / Mass Spectrometers	Tune MSD	As needed	In-House and Outside Vendor
	Change Semi-VOA capillary column	As needed	
	Change Semi-VOA injection port septum	As required	
	Change Semi-VOA injection port liner	As required	
	Replace trap (VOA)	As required	
	Clean ion source	As required	
	Change filament	As required	
	Change electron multiplier	As required	
	Vacuum System: • Mechanical pumps: change oil, change trap pellets (HP only) • Diffusion pump: check oil, check cooling fan, change oil • Turbo pump	• Check every 6 months, check level monthly, change at least annually or sooner is necessary • As required • Replace as required	In-House
	Air Preconcentrators / Autosampler: • Change traps • Inspect Rotors • Calibrate Mass Flow Controllers	• As required • As required • Every 6 months	In-House

Instrument	Applicable Activity	Frequency	Performed
HPLC	Replace/clean check valve filter	As required	In-House
	Replace lamp UV/vis detector	As required	
	Replace flow cell	As required	
	Check flow	Quarterly	
Analytical Balances	Clean pan and compartment	Prior to and after use	In-House and Outside Vendor
	Check with NIST traceable weights	Prior to use	
	Field service	Annually	
Refrigerators and Freezers	Monitor Temperature	Daily	In-House
	Adjust Temperature	As required	
	Clean, Defrost	As required	
Ovens	Clean	As needed or if temperature is outside limit	In-House
pH probes	Condition probe	When fluctuations occur	In-House
	Change Filling Solution	Weekly	
Fluoride ISE	Store in storage solution	Between uses	In-House
Ammonia ISE	Store in storage solution	Between uses	In-House
UV-visible Spectrophotometer	Wavelength check	Annually	In-House
Ion Chromatographs	Change column bed supports	Monthly or as needed	In-House
	Clean column	Monthly or as needed	
	Change column	Every six months or as needed	
	Change valve port face and hex nut	Every six months or as needed	
	Clean valve slider	Every six months or as needed	
	Change tubing	Annually or as needed	
	Eluent pump	Annually	
Restek Thermal Gas Purifier	Check getter tube	Monthly, change as required	In-House

17) Control of Records

17.1 Documentation

ALS Environmental maintains a records system which ensures that all laboratory records of analysis data are retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. Archival procedures are described in the *SOP for Data and Record Archiving* (ADM-ARC).

17.1.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes, but is not limited to, the following items (where applicable) for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes, duplicates and reruns;
- Applicable standard identification numbers;
- Chain of custody, service request and sample acceptance check forms;
- Initial calibration and data review checklist(s);
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

17.2 Information Technology

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. ALS Environmental management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies.

17.2.1 Software Quality Assurance

Practices are defined for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the *SOP for Software and Data Quality Assurance* (ADM-SftwreQA).

The purpose of the SOP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of this plan are policies for software validation and control.

17.2.2 IT Support

The local ALS Environmental Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data described in the *SOP for Electronic Data Backup, Archiving, and Restoration* (ADM-DATA_BU). A software inventory is maintained. Additional IT responsibilities are described in the *SOP for Software and Data Quality Assurance* (ADM-SftwreQA).

In addition to the local IT department, ALS Environmental corporate IT provides support for network-wide systems. ALS Environmental also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

17.2.3 Information Management Systems

ALS Environmental has various systems in place to address specific data management needs. The Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system defines sample identification, analysis specifications, and provides a means of sample tracking. This system is used during sample login to generate the internal service request.

Included on the service request is a summary of client information, sample identification, required analyses, work instructions, and deliverable requirements. The LIMS is used to track the status of a sample and is important in maintaining internal chain of custody.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows Server 2008 R2). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats.

17.2.4 Backup and Security

Laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving.

Full backups onto a hard drive are performed on all file server information once per day. In addition, the laboratory's data warehouse located in Canada performs an offsite full backup nightly.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non-ALS Environmental access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. ALS Environmental uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent or comparable service. The external messaging system operates through a single secure gateway. E-mail attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems with Internet access.

18) Audits

Quality audits are an essential part of ALS Environmental-Simi Valley's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

18.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of ALS Environmental-Simi Valley are conducted regularly by various regulatory agencies and clients. Appendix J lists the certification and accreditation programs in which ALS Environmental-Simi Valley participates. Programs and certifications are added as required. Additionally, internal system audits of ALS Environmental-Simi Valley are conducted regularly under the direction of the QA Manager. The internal audit procedures are described in the *SOP for Internal Audits* (CE-QA001). The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that all elements of the ALS Quality System are assessed.
- Technical/method audits
- Hardcopy report audits

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

18.2 Performance Audits

ALS Environmental-Simi Valley also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. General procedures for these analyses are described in the *SOP for Proficiency Sample Testing Analysis* (CE-QA006). ALS Environmental-Simi Valley routinely participates in the following studies:

- American Industrial Hygiene Association (AIHA) PT Program, 4 per year
- Air and Emissions PT studies, 2 per year
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the QA Manager, Laboratory Director, the laboratory staff, and the Manager of Quality Assurance, USA. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformance (NCAR) procedures.

19) Management Review

Quality assurance requires an active, ongoing commitment by ALS Environmental personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) may also be initiated. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed.

It is the responsibility of each laboratory unit to provide the reporting department with reviewed data accompanied by signature approval. The data validation coordinators provide the Project Manager with a final report of the data. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate Project Manager, who in turn reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative is written (or approved) by the Project Manager to explain any unusual problems with a specific analysis or sample, etc.

The QA Manager provides overview support to the Project Managers as required (e.g., contractually specified, etc.). The QA Manager is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QA Manager regularly communicates with the Laboratory Director to review the various QA/QC activities, priorities, and status of program implementation; including such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

An annual management review of the quality and testing systems is performed as described in the *SOP for Laboratory Management Review* (CE-QA005). This is done to identify any necessary changes or improvements to the quality system or quality assurance policies. This review is documented in a Managerial Review of the Laboratory's Quality Systems and Testing Activities and sent to senior management.

20) Personnel

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at ALS Environmental are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at ALS Environmental when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at ALS Environmental. Safety training begins with reading the *Environmental Health and Safety Manual.* Employees are also required to participate in periodic safety training performed by the Environmental, Health and Safety Coordinator.

Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s). Quality Systems training begins with Quality Assurance orientation for new employees and reading the Quality Assurance Manual. During the employee's first month, the employee receives Ethics training and learns about ALS Environmental quality systems. Each employee participates in annual Ethics Refresher training.

ALS Environmental also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The corporate, company-wide training and development program, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained by the QA department. Training requirements and its documentation are described in the *SOP for Training Policy* (CE-QA003). A training plan is developed whenever an employee starts a new procedure or new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

20.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.
- Where one of the three above is not possible training is performed and supervisor approval is documented.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 20-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

20.2 Continuing Demonstration of Proficiency

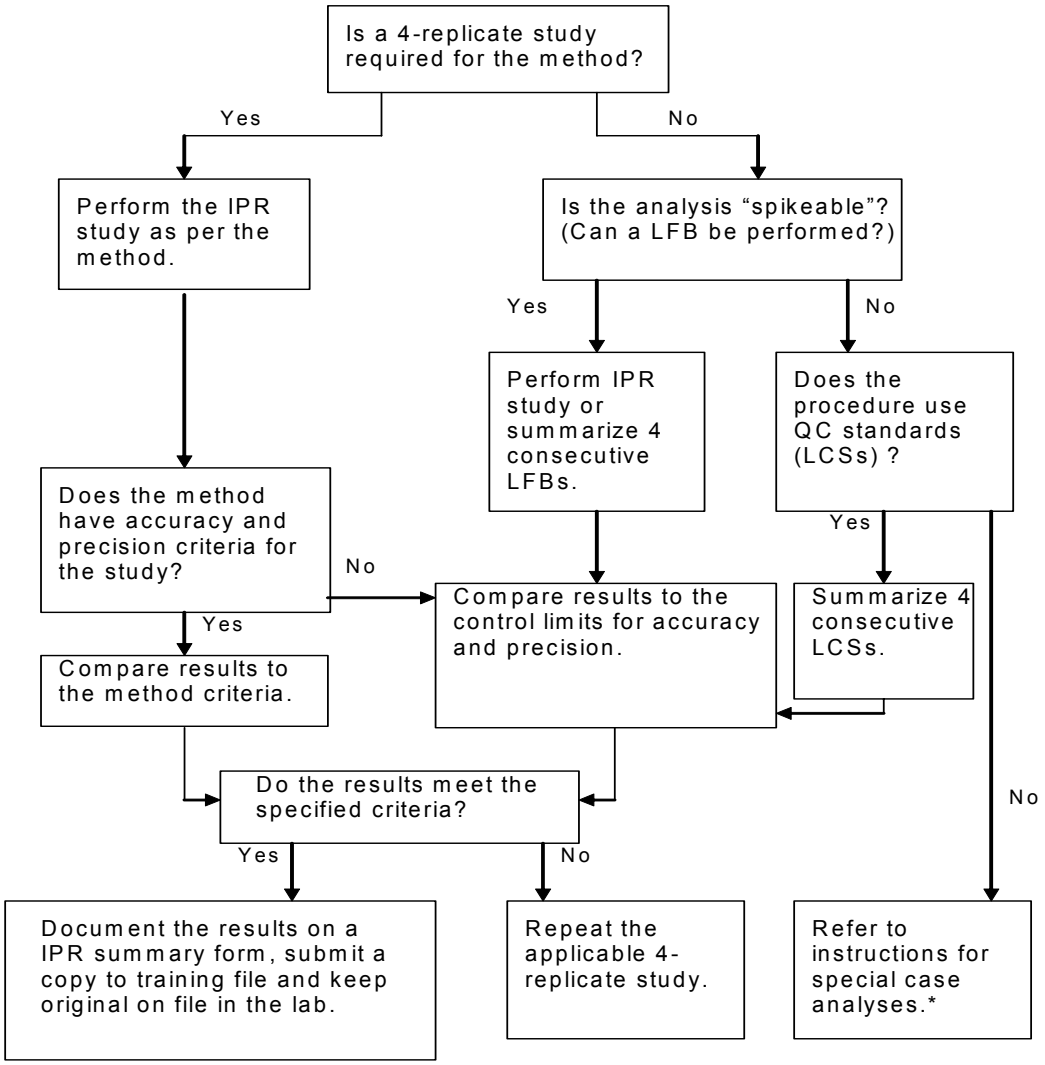
A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

20.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and ALS Environmental resumes. QA maintains a database to record the various technical skills and training acquired while employed by ALS Environmental. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Training Policy* (CE-QA003).

Figure 20-1
Initial Demonstration of Capability Requirements[a]



[a] For IDOC IPR or LFB studies, "second-source" reference materials are used, as per TNI/NELAP requirements
* Refer to the SOP for Training Policy for details. References for Quality Systems, External Documents, Manuals, Standards, and Analytical Procedures

21) Reporting of Results

ALS Environmental reports the analytical data produced in its laboratories to the client via the certified analytical report. This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

21.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). The data is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the supervisor or second qualified analyst reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable it is turned into the reporting department where final reports are generated and then validated by a Data Validation Coordinator. The hardcopy or electronic final report is physically or electronically signed by the project manager and the final report may be stored electronically or in hardcopy format. Test analysis data shall be kept in the appropriate service request folder. Data review and reporting procedures are described in the *SOP for Data Review and Reporting* (ADM-DATA_REV).

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries onto Analytical Records* (CE-QA007).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the "before" and "after" integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration Policy* (CE-QA002).

21.2 Confirmation Analysis

21.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed as required by the method, typically by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.
- The sample meets all of the following requirements:
    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring.

Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.

2. All analytes have been previously analyzed in sample(s) from the same source, identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

21.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
  1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).
  2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS Methods - Two criteria are used to verify identification:
  1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
  2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

21.3 Data Review and Validation of Results

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any

problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.

- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits.

  The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly.
- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. When dilutions are performed the MRL is elevated accordingly.
- Surrogate Results (Organic) – The percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present.

  If no matrix interferences are present and there is no cause for the outlier, the sample is reanalyzed. However, if the recovery is above the upper control limit

with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.

- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used and the results are compared to the MRL. The samples and duplicates are reanalyzed and if re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples reanalyzed or the data reported with the appropriate qualifiers.
- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

21.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package will undergo a peer review by a trained chemist. Prior to release of the report to the client, the Project Manager reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw test data, along with a copy of the final report, is retained by service request number for archival purposes. ALS Environmental maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data is calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Review and Reporting* (ADM-DATA_REV) addresses the flagging and qualification of data. The ALS Environmental-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the analyst or project manager to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Manager verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were

performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Manager accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the ALS Environmental client.

21.5 Deliverables

In order to meet individual project needs, ALS Environmental provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 21-1. Variations may be provided based on client or project specifications.

When requested, ALS Environmental provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. ALS Environmental is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the final report for accuracy.

Table 21-1
Descriptions of ALS Environmental Standard Data Deliverables

Tier I. Routine Certified Analytical Report includes the following:

1. Transmittal letter
2. Chain of custody documents and sample/cooler receipt documentation
3. Sample analytical results
4. Method blank results
5. Surrogate recovery results and acceptance criteria for applicable organic methods
6. Dates of sample preparation and analysis for all tests
7. Case narrative - optional

Tier II. In addition to the Tier I Deliverables, this includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Laboratory Control Sample result(s) with calculated recovery and including associated acceptance criteria
4. Case narrative - optional

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this includes the following:

1. Case narrative - required
2. Summary forms for all associated QC and Calibration parameters, with associated control criteria/acceptance limits

Note: Other summary forms specified in QAPPs or project/program protocols, or those related to specialized analyses such as HRGC/MS will be included.

Tier IV. Full Data Validation Package:

1. All raw data associated with the sample analysis, including but not limited to:
   a. Preparation and analysis bench sheets and instrument printouts,
   b. For organics analyses, all applicable chromatograms, spectral, confirmation, and manual integration raw data. For GC/MS this includes tuning results, mass spectra of all positive hits, and the results and spectra of TIC compounds when requested.
   c. QC data,
   d. Calibration data (initial, verification, continuing, etc),
   e. Calibration blanks or instrument blanks (as appropriate to method).
2. If a project QAPP or program protocol applies, the report will be presented as required by the QAPP.

22) Summary of Changes and Document History

Revision Number	Effective Date	Document Editor	Description of Changes
29	05/30/15	C. Humphrey	2.1 – Revised and added company website
			3.1 – Revised to update NELAC references to NELAP
			3.2 – Revised to update NELAC references to NELAP
			3.8 – Revised to update NELAC references to NELAP
			3.8 – Added Procurement Manager
			Table 3-1 – Added LIMS Manager and Procurement Manager. Updated years of experience.
			12 – Revised to be inclusive of all Method Update Rules and Supplements
			21.1 – Reworded to include both electronic and hardcopy data reduction and review procedures.
			23 – Updated AIHA Policy Modules reference Appendix A – Removed 'NELAC' from glossary
			Appendix B – Updated organization charts
			Appendix E – Updated
			Appendix G – Updated
			Appendix I – Updated
			Appendix J - Updated

23) References for Quality System Standards, External Documents, Manuals, and Test Procedures

The analytical methods used at ALS Environmental generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at ALS Environmental are taken from the references listed below. Additional QA program documents are listed in Appendix I.

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- 2009 TNI Standards.
- American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E).
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 4.2, 10/25/2010.
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 5.0, July 2013.
- American Industrial Hygiene Association-LAP, LLC Policy Document Modules (2A Revision 14, Effective July 1, 2015; 2B Revision 13, Effective July 1, 2015; 6 Revision 3, Effective July 1, 2015), Appendix G (Revision 4, Effective July 1, 2015), and Appendix H (Revision 3, Effective July 1, 2015).
- 3M Organic Vapor Monitor Sampling and Analysis Guide, *Organic Vapor Monitors 3500/3510 and Organic Vapor Monitors 3520/3530*, Technical Bulletin 1028, January 1, 2004.
- 40 CFR Part 60, Test Methods for Standards of Performance for New Stationary Sources, Appendix A.

- 40 CFR Part 63, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Appendix A.
- 40 CFR Part 63, National Emission Standards for Hazardous Air Pollutants for Source Categories, Subchapter C.
- 40 CFR Part 136, Definition and Procedure for the Determination of the Method Detection Limit, Appendix B
- American Society for Testing and Materials (ASTM), Gaseous Fuel, Coal and Coke, Volume 05.06, September 2006.
- American Society for Testing and Materials (ASTM), Annual Book of ASTM Standards, Philadelphia, PA.
- Arizona Administrative Code, *Department of Health Services – Laboratories*, Title 9, Ch. 14, Article 6. *Licensing of Environmental Laboratories*, R9-14-601 through R9-14-621, December 31, 2006 (Supp. 06-4)
- California Environmental Protection Agency Air Resources Board, *Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources*, Volume 3, July 28, 1997.
- California Code of Regulations (CCR), Title 22, Chapter 11 *Identification and Listing of Hazardous Waste*, 7/20/05.
- Minnesota Administrative Rules, *Department of Health*, Chapter 4740, Laboratories; Accreditation Requirements.
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- Environmental Protection Agency, Methods Update Rule (MUR), Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40 CFR Parts 122, 136, 143, 430, 455 & 465; Final Rule 3/12/07, Effective April 11, 2007.
- Environmental Protection Agency, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, Third Edition, 1986 and Updates I (7/92), II (9/94), III (12/96), IIIA (4/98), IIIB (11/04), IVA & IVB. See Chapters 1, 2, 3, 4, 5, 6, and 8.
- Environmental Protection Agency, *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, 1983.
- Environmental Protection Agency, *Methods for the Determination of Inorganic Substances in Environmental Samples,* EPA 600/R-93-100, August 1993.
- Environmental Protection Agency, *EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, Second Edition, EPA/625/R-96-010b, January 1999.
- Environmental Protection Agency, *EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, Second Edition Addendum, October 4, 2000.
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994); 1st Supplement Publication 96-135, 2nd Supplement Publication 98-119, 3rd Supplement 2003-154
- National Council for Air and Stream Improvement, Inc. (NCASI). 2007. *Appendix E - Technical Bulletin Cross Reference Guide for NCASI Methods.* Methods Manual (05).

- *SKC 575 Series Passive Sampler Rate/Selection Guide,* Form #37021, Rev 0012.
- *Standard Methods for the Examination of Water and Wastewater*, 20th Edition (1998).
- South Coast Air Quality Management District, *Laboratory Methods of Analysis for Enforcement Samples.*
- U.S. Department of Labor, Occupational Safety and Health Administration *OSHA Analytical Methods Manual.*

APPENDIX A – Glossary

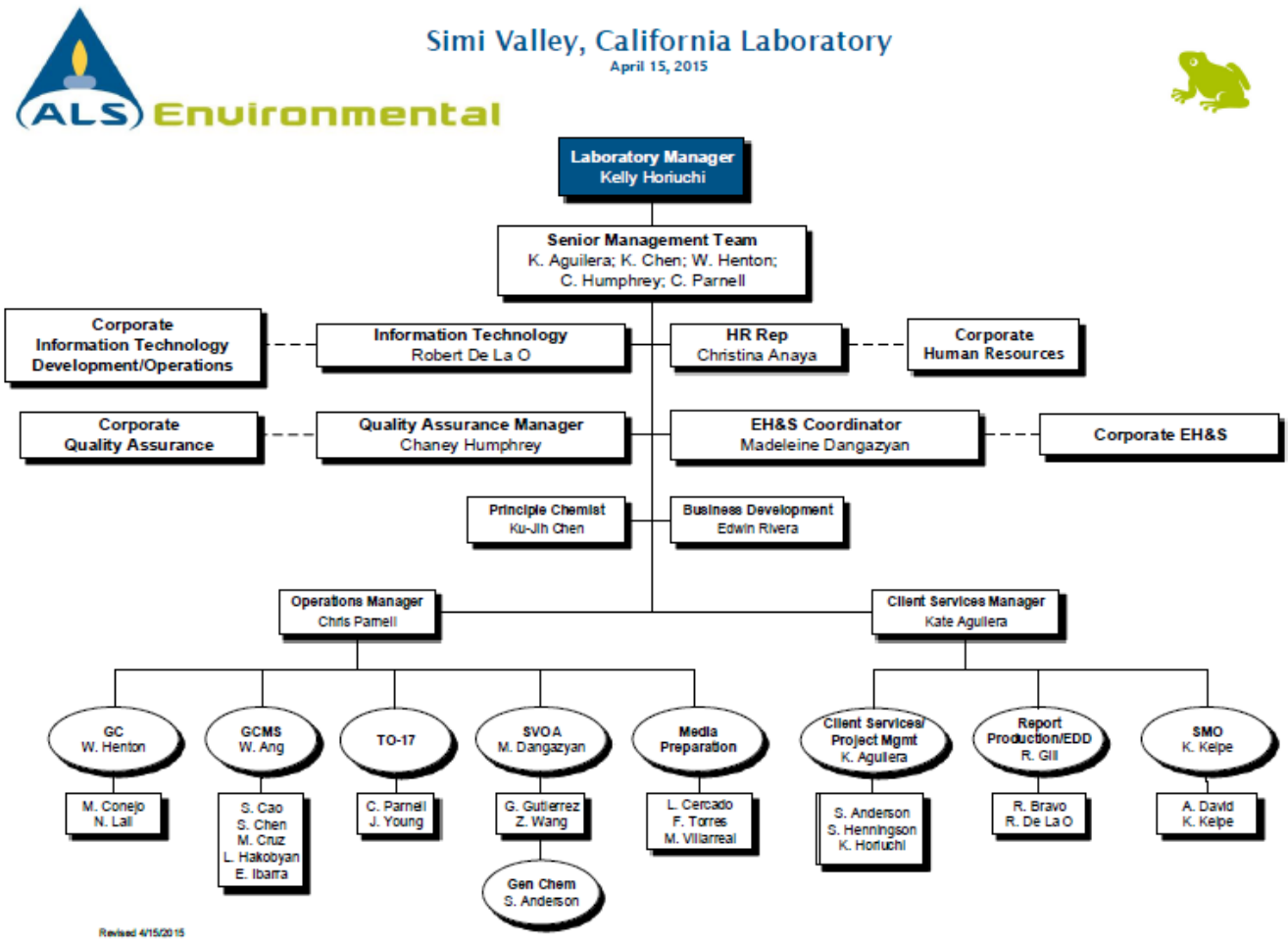
Acronym	Definition
AB	Accrediting Body
ACS	American Chemical Society
ANSI	American National Standards Institute
ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
BFB	4-Bromofluorobenzene
BTEX	Benzene, Toluene, Ethylbenzene, Xylenes
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service Registry Number
CCB	Continuing Calibration Blank sample
CCC	Continuing Calibration Check sample
CCV	Continuing Calibration Verification sample
CDC	Ongoing Demonstration of Capability
CLP	Contract Laboratory Program (through USEPA)
COC	Chain-of-Custody
DCM	Dichloromethane (aka Methylene Chloride)
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOC	Demonstration of Capability
DOE	Department of Ecology (state or federal)
DOH	Department of Health
EPA	U.S. Environmental Protection Agency (aka USEPA)
EPCRA	Emergency Planning & Community Right-to-Know Act
ERA	Environmental Resource Associates
ELAP	Environmental Laboratory Accreditation Program
FID	Flame Ionization Detector
FIFRA	Federal Insecticide, Fungicide & Rodenticide Act
FR	Federal Register
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HP	Hewlett-Packard (mfg. GC instruments)

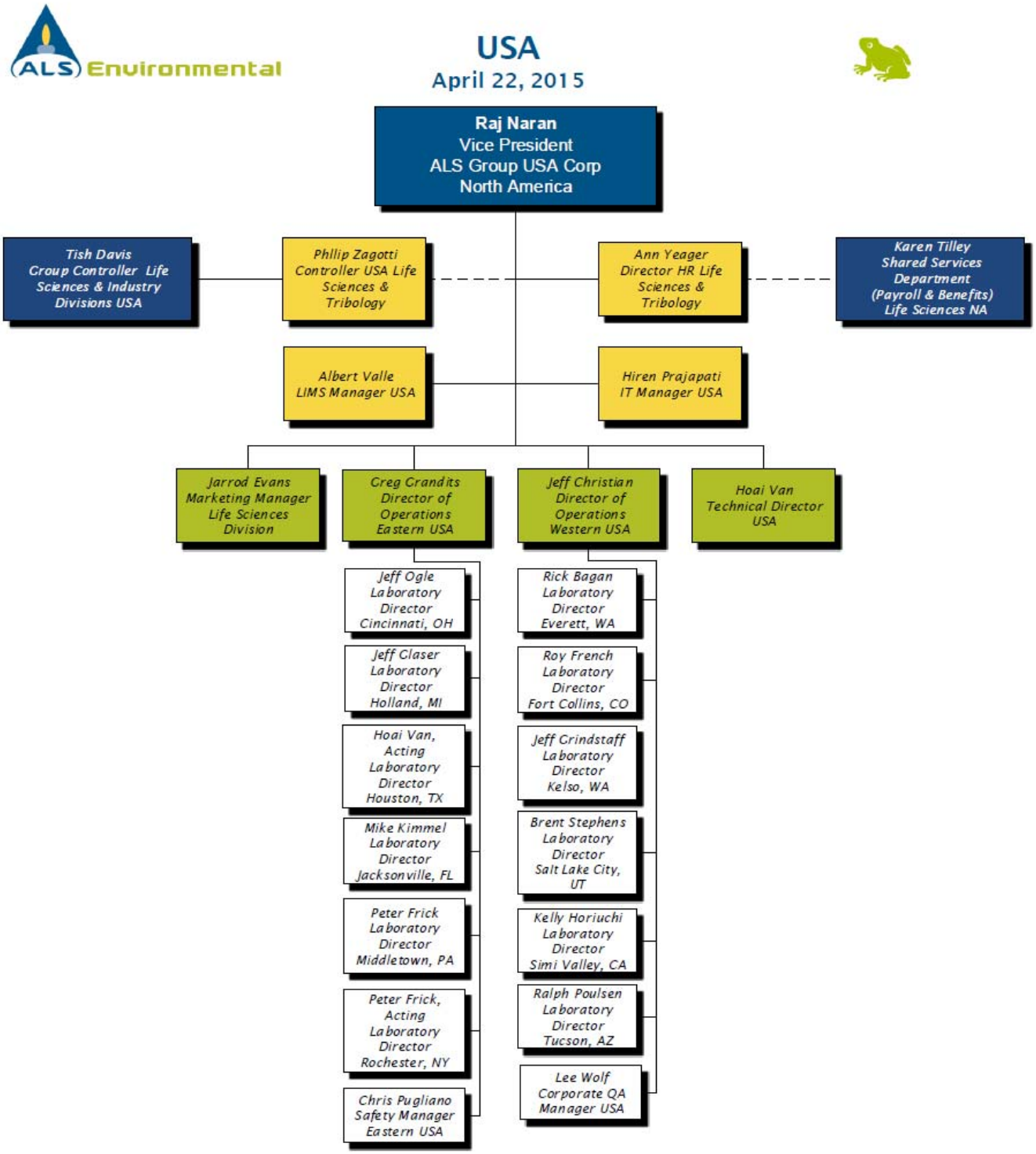
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICAL	Initial Calibration
ICB	Initial Calibration Blank sample
IDC	Initial Demonstration of Capability
ICV	Initial Calibration Verification sample
IFB	Invitation for Bid
ISO/IEC	International Organization for Standardization/International Electrochemical Commission
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LUFT	Leaking Underground Fuel Tank
MB	Method Blank
MDL	Method Detection Limit
MRL	Method Reporting Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NAS	National Academy of Sciences
NELAP	National Environmental Laboratory Accreditation Program
NCASI	National Council for Air and Stream Improvement (for the Paper Industry)
NCI	National Cancer Institute
ND	Not Detected
NIH	National Institute of Health
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NPD	Nitrogen Phosphorus Detector
NPDES	National Pollutant Discharge Elimination System
NSF	National Science Foundation
NTIS	National Technical Information System
NTP	National Toxicology Program
OSHA	Occupational Safety and Health Administration
PCBs	Polychlorinated Biphenyls
PE	Performance Evaluation sample
PID	Photoionization Detector

PQL	Practical Quantitation Limit
PT	Proficiency Test
QA	Quality Assurance
QAM	Quality Assurance Manual
QC	Quality Control
RAS	Routine Analytical Services (Contracts through USEPA)
RCRA	Resource Conservation and Recovery Act
RFP	Requests for Proposal
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SAS	Special Analytical Services (contracts through USEPA)
SIE	Selective Ion Electrode
SIM	Selected Ion Monitoring
SMO	Sample Management Office (aka Sample Receiving)
SOC	Semi-Volatile Organic Compounds
SOP	Standard Operating Procedure
SOQ	Statement of Qualifications
SOW	Statement of Work
SVOAs	Semi-Volatile Organic Analytes
SVOCs	Semi-Volatile Organic Compounds
SW-846	Test Methods for Evaluating Solid Waste, Physical/Chemical Methods
TNI	The NELAC Institute
TPH	Total Petroleum Hydrocarbons
TSCA	Toxic Substances Control Act
UST	Underground Storage Tank
UV	Ultraviolet Spectrophotometer
VOA	Volatile Organic Analyte
VOC	Volatile Organic Compounds
WP	Water Pollution
WS	Water Supply

Units	Definition
mg/kg	Milligrams per Kilogram
mg/L	Milligrams per Liter
mg/m3	Milligrams per Cubic Meter
ng/L	Nanograms per Liter
ppb	Parts Per Billion
ppbV	Parts Per Billion Volume
ppm	Parts Per Million
ppmV	Parts Per Million Volume
ug/L	Micrograms per Liter
ug/m3	Micrograms per Cubic Meter

APPENDIX B – Organization Charts and Key Personnel Qualifications





Kathleen 'Kate' Aguilera

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ALS Environmental

Education

California State University - Northridge, CA
BA, Chemistry, 1989

Affiliations

American Chemical Society

Client Services Manager / Project Manager

2011 – Present

Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinates with the laboratory and administration to ensure that analyses are properly executed and meets the clients' needs.

Previous Experience

Columbia Analytical Services, Inc. — **Project Manager, '97 – '11**
Simi Valley, CA

Responsibilities: Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinates with the laboratory and administration to ensure that analyses are properly executed and meets the client's needs.

Columbia Analytical Services, Inc. — **GC/MS Analytical Chemist, '94 – '97**
(DBA Performance Analytical, Inc.)
Los Angeles, CA

Responsibilities: Analysis of air samples using EPA compendium methods TO-1, TO-2 and TO-14 using cryogenic concentration and thermal desorption techniques on whole air samples collected in summa canisters, Tedlar bags, and solid sorbent air samples. Proficient in the interpretation of mass spectra. Responsible for the preparation and quality control verification of solid sorbent sampling media for EPA Compendium methods TO-1 and TO-2.

Performance Analytical, Inc. — **GC/MS Analytical Chemist, '92 – '94**
Canoga Park, CA

Responsibilities: Analysis of air samples using EPA compendium methods TO-1, TO-2 and TO-14 using cryogenic concentration and thermal desorption techniques on whole air samples collected in summa canisters, Tedlar bags, and solid sorbent air samples. Proficient in the interpretation of mass spectra. Responsible for the preparation and quality control verification of solid sorbent sampling media for EPA Compendium methods TO-1 and TO-2.

Performance Analytical, Inc. — **GC Analytical Chemist, '89 – '92**
Canoga Park, CA

Responsibilities: Performed analyses of air samples for reduced sulfur compounds, hydrocarbon distribution and speciation, fixed atmospheric gases and total gaseous non-Methane organics. Performed analyses of soil and water samples for TPHg (mod. 8015) and BTEX. Performed extractions and analyses of CARB, NIOSH, OSHA and EPA 8000 series methods. Also performed metals analysis using flame and graphite furnace atomic absorption spectrophotometry (AA, GFAA).

Susan 'Sue' Anderson

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ALS Environmental

Education

University of Illinois - Urbana-Champaign, IL
BS, Biochemistry, 1989

Project Manager/Technical Manager (General Chemistry)

2011 – Present

Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinates with the laboratory and administration to ensure that analyses are properly executed and meets the clients' needs. Also responsible for the training of general chemistry staff, maintenance of MDL studies, and standard operating procedures, data evaluation and report responsibility.

Previous Experience

Columbia Analytical Services, Inc. — **Project Manager/Technical Manager (General Chemistry), '06 – '11**
Simi Valley, CA

Responsibilities: Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinates with the laboratory and administration to ensure that analyses are properly executed and meets the client's needs. Also responsible for the training of general chemistry staff, maintenance of MDL studies, and standard operating procedures, data evaluation and report responsibility.

Columbia Analytical Services, Inc. — **Project Manager/Technical Manager (General Chemistry), '02 – '06**
Canoga Park, CA

Responsibilities: In addition to the Project Manager duties listed below, also responsible for the management of General Chemistry laboratory operations, including the financial aspects. This includes supervision and coordination of work load and training personnel as necessary as well as supervision of method development and certification, maintenance of MDL studies and SOPs, data evaluation and report responsibility. Other duties include participation in the formulation of project strategy and meetings involving major technical issues, working with regional senior management in short and long-range planning, and other duties as assigned.

Columbia Analytical Services, Inc. — **Project Manager II, '00 – '02**
Canoga Park, CA

Responsibilities: Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling and tracking from the delivery of sample bottles to client site to the delivery of the completed analytical report. Ensures that the client receives timely, appropriate, and quality analytical services. Coordinates with the CAS laboratory and administration to ensure that analyses are properly executed and meet the clients' needs. Coordinates sub-contracting with internal and external laboratories. Acts as a liaison for all client-related activities within Columbia Analytical Services, Inc. Interfaces with work processing staff to answer technical questions that arise during EDD completion. Has high level role in data evaluation and report responsibility. High level client and regulatory agency contact.

Columbia Analytical Services, Inc. — **Scientist I-III, '92 – '00**
Canoga Park, CA

Responsibilities: Responsible for performing inorganic analyses such as: alkalinity, ammonia, BOD, COD, cyanide, sulfide, reactivity, fluoride, pH, hardness, hexavalent chromium, phenols, surfactants, total-dissolved-suspended solid, conductivity, turbidity, nitrate, chloride by titration, turbidimetric sulfate, color, odor, organic lead, residual chlorine, settleable solids, specific gravity, carbon dioxide, TCLP/STLC metals and semi-volatile extraction. Also perform analyses for TRPH and oil and grease and occasionally perform metals digestion. Also ran the Graphite furnace for all furnace metals and was responsible for standard prep and maintenance.

National Environmental Testing — **Wet Chemistry, '90 – '91**
Bartlett, IL

Responsibilities: Responsible for the analyses for wastewater parameters and some inorganic analytes.

Widayati 'Wida' Ang

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ALS Environmental

Education

Technical University of West Berlin -
West Berlin, Germany
BS, Chemistry 1982

Technical University of West Berlin -
West Berlin, Germany
MS, Chemistry 1984

Volatile GC/MS Team Leader

2011 – Present

Team leader for the Volatile Gas Chromatography Mass Spectrometry Air group responsibilities are but are not limited to training of chemists, peer review of analytical data, mentoring of junior analysts, standard operating procedure review and streamlining of methods. Duties also require performance reviews and development of her direct reports.

Previous Experience

Columbia Analytical Services, Inc. — **Volatile GC/MS Team Leader, '08 – '11**
Simi Valley, CA
Duties as above.

Columbia Analytical Services, Inc. — **GC/MS Chemist, '07 – '08**
Simi Valley, CA
Analyzing indoor air, ambient air and source emission samples by GC/MS methods, standard preparation, perform maintenance on instruments when required, real time data reduction, participate in peer review process, and good practice of all QA/QC requirements.

Columbia Analytical Services, Inc. — **Technical Manager, Organic Chemistry, '99 – '07**
Canoga Park, CA
Responsible for managing the organics department with regards to State and Federal regulatory requirements. Supervises and coordinates work load and trained personnel. Supervised method development and certification, as well as method troubleshooting and instrument maintenance. Responsible for mobile laboratory operations.

Laboratory Data Consultants, Inc. — **Data Validator, '98 – '99**
Carlsbad, CA
Responsible for retrieving analytical data from closed down laboratory operations, review and validation of data packages. Supervised other employees for data package assembly

VOC Laboratories, Inc. — **Assistant QC Manager and Data Package Specialist, '96 – '98**
Glendale, CA
Responsible for overseeing data quality of final data validation packages. Managed production of data packages to meet various State and Federal analytical programs as well as customized client formats. Oversaw enforcement of the laboratory for implementation of corrective action measure. Interacted with chemists and project managers to ensure accuracy and completeness of data deliverables.

Thermo Analytical — **Technical Director/Department Manager, '92 – '96; Department Supervisor and Chemist, '88 - '92**
Monrovia, CA
Responsible for daily operations of the organic chemistry department. Developed standard operating procedures for various methods. Reviewed analytical data generated for completeness and contractual requirements according to Contract Laboratory Program (CLP) and SW-846 methods. Organized and scheduled reports for project managers. Responsible for upgrading and purchasing new instrumentation. Provided technical support to QC coordinator and laboratory personnel. Assisted with proposal preparation and audits. Responsible for training chemists and technicians in proper performance of various analytical methods. Responsible for sample analysis of water, soil, and air for volatile organics by GC and GC/MS. Assisted chemists in the analysis and interpretation of pesticides and PCBs.

Shankman Laboratories — **Analytical Chemist, '86-'88**
Los Angeles, CA
Prepared and analyzed soil and water samples using GC, GC/MS, HPLC, IR, IC and UV spectrophotometic techniques

Ku-Jih Chen

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ALS Environmental

Education

National Chung-Hsing University - Taipei, Taiwan
BS, Botany, 1975

Principle Chemist

2011 – Present

Responsible for the development and validation of sampling and analysis methods, new technology and laboratory automation.

Previous Experience

Columbia Analytical Services, Inc.
Simi Valley, CA
Principle Chemist, '00 - '11
Responsibilities: Responsibilities listed above.

Columbia Analytical Services, Inc.
(dba Performance Analytical, Inc.)
Los Angeles, CA
Scientist VII, '94 - '00
Responsibilities: Responsibilities included operating the gas chromatography and sample preparation laboratories, developing methods (previously developed the Total Combustion Analyzer for the measurement of reactive organic gases in stationary source samples, and the Determination of Reduced Sulfur Compounds and fixed atmospheric gases in POTW emissions, refinery and landfill gases), and serving as the laboratory's primary Industrial Hygiene Chemist.

Performance Analytical, Inc.
Canoga Park, CA
Principle Chemist, '89-'94
Responsibilities: Responsibilities listed above.

C-E Environmental, Inc.
Camarillo, CA
Extraction Laboratory Supervisor, '84 – '89
Responsibilities: Responsibilities included supervising chemists, associate chemists, and technicians, preparing SOPs, analytical standards, and spiking solutions, serving as Primary Extraction Chemist for the Love Canal Habitability Study, and previously responsible for instrumental analysis using GC, LC, GC/MS, and AA.

Paolyta Company
Taipei, Taiwan
Research and Development Chemist, '80 – '84

Panlabs Taiwan Ltd.
Taipei, Taiwan
Research Chemist, '75 – '80

Madeleine Dangazyan

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ALS Environmental

Education

California State University at Northridge - Northridge, CA
BS, Chemistry 1995

Semi-Volatiles Technical Manager and EH&S Manager

2011 – Present

As EH&S Manager, is responsible for the implementation of the Environmental Health and Safety program of ALS North America to this facility. Duties include accident investigation and incident review, maintenance of all safety-related equipment and documents, and performing safety audits and reporting results to management. Semi-Volatiles/Industrial Hygiene Technical Manager responsibilities are but not limited to training of junior chemists, data reduction and peer review of analytical data, mentoring of junior analysts, writing and reviewing of standard operating procedures. Development and implementation of new methods. Duties also require performance reviews and development of direct reports. Additional responsibilities are analyzing ambient air, source emissions, and industrial hygiene samples using GC and HPLC utilizing OSHA, NIOSH and EPA mandated methodologies. Preparation and analysis of air samples taken on various sorbent tubes for semi-volatile organic compounds. Determination of Carbonyls, Phenols and Cresols in ambient air and source emission samples using HPLC. Determination of Polynuclear Aromatic Hydrocarbons using EPA Method TO-13A. Analysis of Pesticides and PCBs using EPA Methods TO-4A and TO-10A. Routine and necessary instrument maintenance.

Previous Experience

Columbia Analytical Services, Inc. — **Semi-Volatiles Technical Manager, '02- '11; EH&S Manager '10-11**
Simi Valley, CA

Responsibilities: Responsibilities listed above.

Columbia Analytical Services, Inc. — **Scientist, '00- '02**
Simi Valley, CA

Responsibilities: Responsibilities include training of chemists, peer review of analytical data, mentoring or junior analyst, standard operating procedure review, and streamlining of methods. Additional responsibilities are analyzing ambient air, source emissions, and industrial hygiene samples using GC and HPLC utilizing OSHA, NIOSH and EPA mandated methodologies. Preparation and analysis of air samples taken on various sorbent tubes for semi-volatile organic compounds. Determination of Carbonyls, Phenols and Cresols in Aromatic Hydrocarbons using EPA Method TO-13A. Analysis of Pesticides and PCBs using EPA Methods TO-4A and TO-10A. Routine and necessary instrument maintenance.

Columbia Analytical Services, Inc. — **Scientist, '99- '00**
(dba Performance Analytical, Inc.)
Simi Valley, CA

Responsibilities: Responsibilities include analyzing indoor and ambient air, source emission, and industrial hygiene samples by GC methods.

Air Products and Chemicals, Inc. — **Analytical Chemist, '95 – '99**
Long Beach, CA

Responsibilities: Quality assurance analysis of EPA protocol gases utilizing GC, FTIR and NDIR. Preparation of personnel schedules, lead laboratory contact.

California State University at Northridge — **Undergraduate Researcher, '93 – 94**
Northridge, CA

Responsibilities: Assisted professor with improving and implementing student laboratory experiments to better utilize a GC/MS.

Wade Henton

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ALS Environmental

Education

University of California at Santa Barbara – Santa Barbara, CA
BS, Chemistry 1985

Volatile GC Team Leader

2011 – Present

Team leader for the Volatile Gas Chromatography department where responsibilities include but are not limited to training of chemists, peer review of analytical data, mentoring of junior analysts, standard operating procedure review, and streamlining of methods. Duties also require performance reviews and development of direct reports.

Previous Experience

Columbia Analytical Services, Inc. — **Volatile GC Team Leader, '00 – '11**
Simi Valley, CA
Responsibilities listed above.

Columbia Analytical Services, Inc. — **Scientist V, '95 – '00**
(dba Performance Analytical, Inc.)
Los Angeles, CA
Responsibilities include analyzing indoor and ambient air, source emission, and industrial hygiene samples by GC and GC/MS methods.

Columbia Analytical Services, Inc. — **Scientist IV, '94 – '95**
(dba Performance Analytical, Inc.)
Los Angeles, CA
Responsibilities listed above.

Coast-to-Coast Analytical Services — **Analytical Chemist, '92 – '94**
Camarillo, CA
Responsibilities included analyzing samples using EPA methods 625, 525 and 1625 as well as developing new methods for GC/MS testing.

Coast-to-Coast Analytical Services — **Analytical Chemist, '91 – '92**
Goleta, CA
Responsibilities included analyzing samples using EPA methods 624 and 524.2 by GC/MS. Used GC/MS methods to perform fuel fingerprinting

Combustion Engineering Environmental — **Analytical Chemist, '86 – '91**
Camarillo, CA
Responsibilities included method development for GC and HPLC. Analysis of samples using EPA methods 608, 615, 631, 632 and SW846. Other methods used include 8080, 8010, 8020, 8150 and 8030. Oversaw data integrity for the GC Laboratory instrument data network. Data review.

Fortin Industries — **Chemist, '86**
Sylmar, CA
Research and Development and Quality Assurance/Quality Control on polymer products and metal coatings using differential scanning calorimeters, scanning electron microscope, AA, GC, and HPLC.

Kelly M. Horiuchi

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ALS Environmental

Education

California State University, Northridge,CA
BA, Biology, 1998

Laboratory Director

2011 - Present

Primary responsibilities include management of all laboratory departments, scheduling, productivity, reporting and evaluation of analytical methodologies, project planning, budgeting, and Quality Assurance/Quality Control protocol oversight. Other responsibilities include conducting facility compliance reviews; providing departmental support for equipment purchases; resolving personnel issues; determining resource allocation; and providing supervision, training, and leadership to key laboratory staff. In addition, other responsibilities include direct responsibility for national contracts and consultants.

Previous Experience

Columbia Analytical Services, Inc.
Simi Valley, CA
Laboratory Director, '09-'11

Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance.

Columbia Analytical Services, Inc.
Simi Valley, CA
Project Manager, '05-'09

Responsibilities: Interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinated with the laboratory and administration to ensure that analyses were properly executed and meets the client's needs.

Columbia Analytical Services, Inc.
Simi Valley, CA
Data Validation Coordinator,'03-'05

Responsibilities: Validation of analytical results produced by the laboratory. Verification of client analytical requests, sample information, and reporting formats. Interacts with project managers and Quality Assurance Program Manager to ensure that all reports fulfill client requirements as well as QA/QC needs. Compiled quality control summary, and calibration data upon client request for data packages. Assist the Quality Assurance Program Manager with standard operating procedures, control charting, and audit preparation.

Cure Autism Now
Los Angeles, CA
Database Analyst, '02-'03

Responsibilities: Performed analysis of test date through data audits and queries, maintained extensive database, and coordinated data audits between Northern and Southern California locations. Additional duties included assisting in the creation of new databases, as needed, creation of SOP for phenotypic and genotypic data collecting, and process improvements for subject flow through the research project.

Columbia Analytical Services, Inc.
(dba Performance Analytical, Inc.)
Simi Valley, CA
Scientist II, Data Validation Coordinator, '00-'02

Responsibilities: Validation of all analytical results produced by the laboratory. Verification of client analyses, sample information, and reporting format. Compiled quality control summary, and calibration data upon client request for data packages. Assisted the Quality Assurance Program Manager with standard operating procedures, control charting, and audit preparation.

Specialty Laboratories
Santa Monica, CA
Administrative Assistant, Data Analyst, '99-'00

Responsibilities: Performed retrieval, quality control, and organization of data. Compiled data for reporting of HIV, lead, urinalysis, kidney stones, and communicable diseases. Also communicated with the state DOH and clients regarding reporting requirements and demographic information.

Chaney Humphrey

2655 Park Center Drive, Suite A • Simi Valley, CA 93065 • +1 805 526 7161

ALS Environmental

Education

Oregon State University,
Corvallis, OR
BS, Biology, 2004

Quality Assurance Manager

2011 - Present

Responsibilities include facilitate ethics and QA training, maintain all training documentation, perform QA orientation for new employees, review data (both hardcopy and electronic), perform internal QA audits and prepare written reports, review, approve, and control Standard Operating Procedures, maintain QA Manual, maintain QA records (including archived logbooks, archived certificates of analysis, nonconformity and corrective action reports, MDL studies results, SOP revision and distribution, statistical control limits, PE sample results), serve as document control officer, and PC for all PE sample analyses, prepare corrective action report for any unacceptable PE sample results, maintain laboratory's certifications and approvals, facilitator for external QA audits and prepare written response to deficiencies, prepare activity report to management.

Previous Experience

Columbia Analytical Services, Inc. — **Quality Assurance Manager, -09 -11**
Simi Valley, CA

Duties same as above.

Columbia Analytical Services, Inc. — **Data Validation Coordinator, '07-'09**
Simi Valley, CA

Responsibilities include validation of analytical results produced by the laboratory. Verification of client analytical requests, sample information, and reporting formats. Interacts with project managers and Quality Assurance Program Manager to ensure that all reports fulfill client requirements as well as QA/QC needs. Compiled quality control summary, and calibration data upon client request for data packages.

Columbia Analytical Services, Inc. — **GC/MS Chemist,'05-'07**
Simi Valley, CA

Responsibilities: Analyzing indoor air, ambient air and source emission samples by GC/MS methods, standard preparation, perform maintenance on instruments when required, real time data reduction, participate in peer review process, and good practice of all QA/QC requirements.

Columbia Analytical Services, Inc. — **Analyst, '04-'05**
Kelso, WA

Responsibilities: Performed a variety of analytical tests within the General Chemistry laboratory according to EPA Methodologies including Ion Chromatography, total sulfur, and solids. Saturday crew member responsible for performance of all short hold time methods including microbiology methodologies.

Columbia Analytical Services, Inc. — **Temporary Employee, Summers '02-'04**
Kelso, WA

Responsibilities: Temporary employee (summers) performing a variety of analytical tests including grain size, total organic carbon, total suspended solids, total dissolved solids, alkalinity, acidity, and chemical oxygen demand. Additionally, performed colorimetric methods including ortho-phosphorous, total-phosphorous, hexavalent chromium, and nitrite as nitrogen.

Christopher Parnell

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ALS Environmental

Education

University of California
at Santa Barbara
Santa Barbara, CA
BS, Chemistry 1986

Operations Manager/Technical Advisor (Volatile GC/MS Air)

2012 – Present

Operation Managers responsibilities include planning, directing, and coordinating the operations of the laboratory departments. Duties and responsibilities include formulating policies, managing daily operations, and planning the use of materials and human resources. Reviews performance data to measure productivity and goal achievement and to determine areas needing cost reduction and program improvement to increase efficiency.

Technical Advisor for the Volatile Gas Chromatography Mass Spectrometry department. Has the responsibility of oversight of training of chemists, peer review of analytical data, mentoring of junior analysts, standard operating procedure review and streamlining of methods. Duties also require performance reviews and development of direct reports.

Previous Experience

ALS Environmental
Simi Valley, CA
Technical Advisor (Volatile GC/MS Air), '11 – '12
Responsibilities: Technical Advisor responsibilities listed above.

Columbia Analytical Services, Inc.
Simi Valley, CA
Technical Advisor (Volatile GC/MS Air, '08 – '11
Responsibilities: Technical Advisor responsibilities listed above.

Columbia Analytical Services, Inc.
Simi Valley, CA
GC/MS Team Leader, '00 – '08
Responsibilities: Team leader for the Volatile Gas Chromatography Mass Spectrometry group. Responsibilities include training of chemists, peer review of analytical data, mentoring of junior analysts, standard operating procedure review, and streamlining of methods. Duties also require performance reviews and development of direct reports.

Columbia Analytical Services, Inc.
(dba Performance Analytical, Inc.)
Los Angeles, CA
Scientist VI, '94 – '00
Responsibilities: Responsibilities include analyzing indoor air, ambient air and source emission samples by GC/MS methods, standards preparation, perform maintenance on instruments when required, real time data reduction, participation in peer review process, and good practice of all QA/QC requirements.

Performance Analytical, Inc.
Canoga Park, CA
Scientist VI, '91 – '94
Responsibilities: Responsibilities listed above.

ABB Environmental Inc.
Camarillo, CA
Air Toxics Laboratory Supervisor, '90 – '91
Responsibilities: Responsibilities included scheduling client analyses and developing methods for non-routine analyses, and operating the Air Toxics laboratory.

C-E Environmental Inc., EMSI
Camarillo, CA
Analytical Chemist, '87 – '90
Responsibilities: Responsibilities included overseeing the Pesticide/PCB analysis of samples under the EPA Contract Laboratory Program, and interfacing with the EPA and regional offices to respond to inquiries, and performing GC analyses and extractions.

Damon Reference Laboratory
Newbury Park, CA
Chemist, '86 – '87
Responsibilities: Responsibilities included performing Enzyme-linked immunosorbent assays, Western-Blot assays, and Protein Electrophoresis.

APPENDIX C – Ethics and Data Integrity Policy

ETHICS AND DATA INTEGRITY AGREEMENT

I state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at ALS.

I agree that in the performance of my duties at ALS:

1. I shall not intentionally report data values that are not the actual values obtained;
2. I shall not intentionally report the dates, times and method citations of data analyses that are not the actual dates, times and method citations of analyses;
3. I shall not intentionally represent another individual's work as my own;
4. I shall not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operating Procedures, or as defined by company policy.
5. I agree to inform ALS of any accidental or intentional reporting of non-authentic data by other employees.
6. I have read this ethics and data integrity agreement and understand that failure to comply with the conditions stated above will result in disciplinary action, up to and including termination.
7. I agree to adhere to the following protocols and principals of ethical conduct in my work at ALS. All work assigned to me will be performed using ALS approved methods and procedures and in compliance with the quality assurance protocols defined in the ALS Quality System.
8. I will not intentionally falsify nor improperly manipulate any sample or QC data in any manner. Furthermore, I will not modify data values unless the modification can be technically justified through a measurable analytical process or method acceptable to ALS. All such modifications and their justification will be clearly and thoroughly documented in the raw data and appropriate laboratory record, and will include my initials or signature and the date.
9. I will not make false statements to, or seek to otherwise deceive ALS staff, managers or clients. I will not knowingly, through acts of commission, omission, erasure or destruction, improperly report any test results or conclusions, be they for client samples, QC samples, or standards.
10. I will not condone any accidental or intentional reporting of unauthentic data by other ALS staff and will immediately report such occurrences to my Supervisor, Lab Director, Quality Assurance Manager, or Human Resources. I understand that failure to report such occurrences may subject me to immediate discipline, including termination.
11. If a supervisor, manager, director or other member of the ALS leadership group requests me to engage in or perform an activity that I feel is compromising data validity or defensibility, I have the right to not comply with the request. I also have the right to appeal this action through an ALS local Quality Staff, Corporate Quality Assurance or Human Resources.
12. I understand that if my job includes supervisory responsibilities, I will not instruct, request or direct any subordinate to perform any unethical or non-defensible laboratory practice. Nor will I discourage, intimidate or inhibit a staff member who may choose to appropriately appeal my supervisory instruction, request or directive that may be perceived to be improper, nor retaliate against those who do so.
13. I understand that employees who report violations of this policy will be kept free from intimidation and recrimination arising from such reporting.

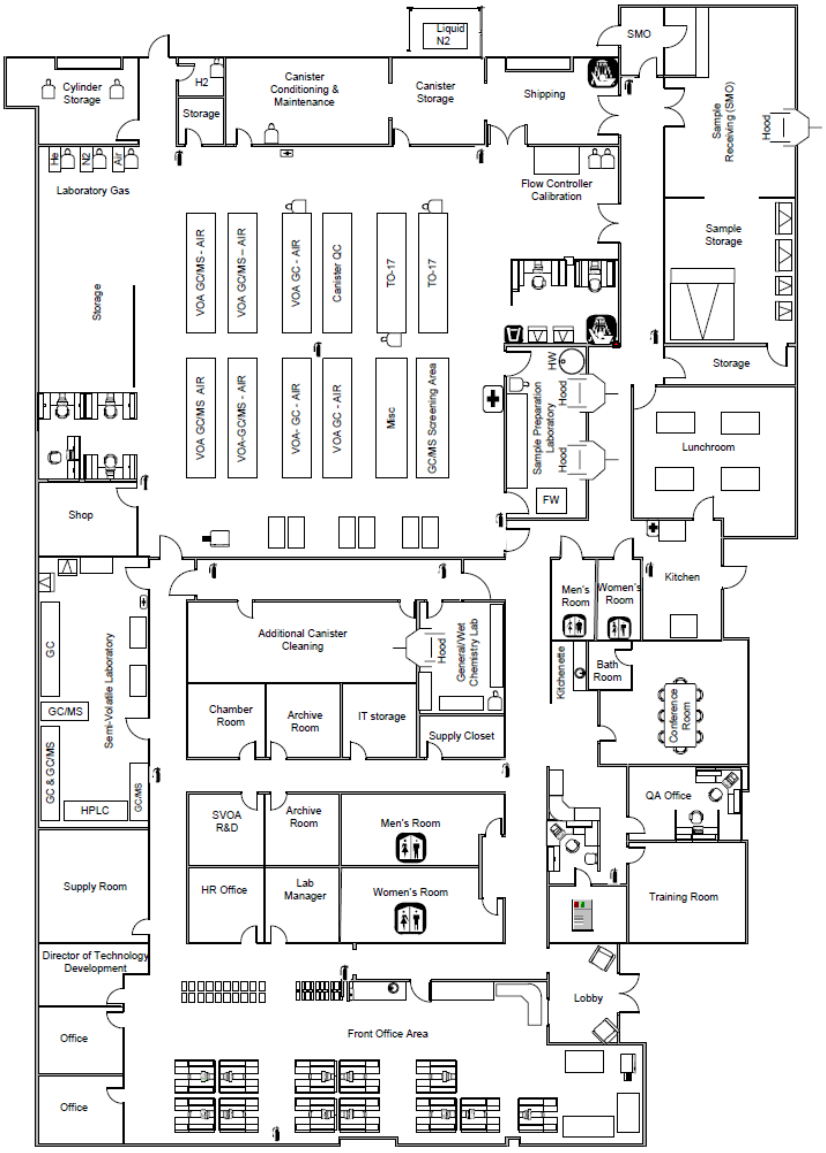
I have read, and understand the above policy and realize that failure to adhere to it may result in disciplinary action, up to and including termination. Compliance with this policy will be strictly enforced with all personnel employed by the company.

Employee Name _____ Signature _____

ALS Location_____ Date _____

APPENDIX D – Laboratory Floor Plan

ALS Environmental-Simi Valley Laboratory Floor Plan

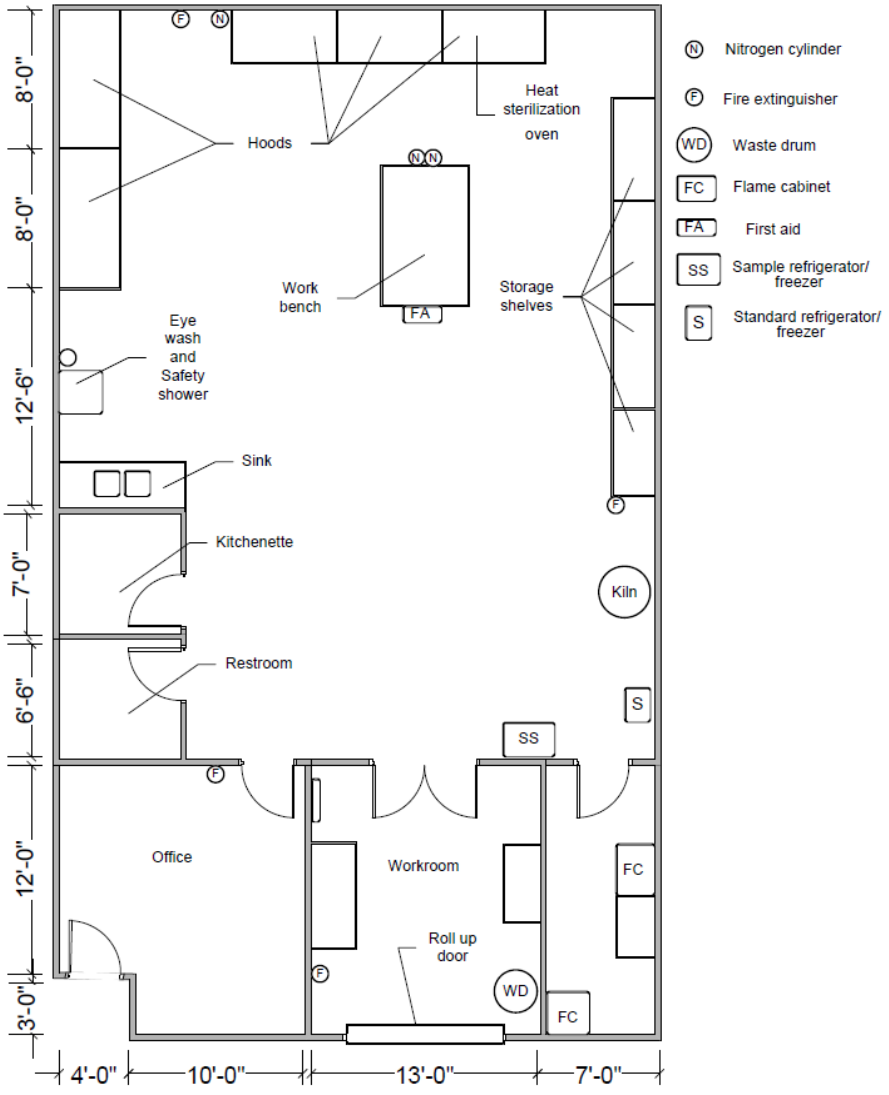


ALS ENVIRONMENTAL - SIMI VALLEY FLOOR PLAN		
2655 Park Center Drive, Suite A, Simi Valley, California 93065		
-First Aid	HW -Hazardous Waste Cabinet	
-Network Server Room	-Emergency Shower	FW -Flammable Waste Cabinet
-Fire Extinguisher	-Gas Cylinder(s)	
-Refrigerator/Freezer	-Deionized Water	

ALS Environmental-Simi Valley Extraction Laboratory Floor Plan

Extraction Laboratory for
ALS Environmental

2360 Shasta Way, Unit G
Simi Valley, CA. 93065



APPENDIX E – Analytical Equipment

Equipment Description - Gas Chromatography	Purchased / Acquired	Location
Screen 02: Hewlett-Packard 5890 with FID Detector	-	VOA GC/MS Screen
Screen 03: Hewlett-Packard 5890 with FID Detector	-	VOA GC/MS Screen
GC01: Hewlett-Packard 5890 with FID/TCD Detectors *Fixed Gas Analyzer/Total Combustion Analyzer (TCA)*	1995	VOA GC
GC03: Hewlett-Packard 5890 with ECD/FID Detectors *Hewlett-Packard 7673 Autosampler*	1995	SVOA
GC05: Hewlett-Packard 5890 Series II Combined with Sievers 355 (SCD 1)	1996	SVOA
GC06: Hewlett-Packard 6890 with ECD/ECD Detectors *Hewlett-Packard 6890 Autosampler*	1995	SVOA
GC07: Hewlett-Packard 6890 with FID/FID Detectors	1995	VOA GC
GC08: Hewlett-Packard 5890 Series II with TCD/FID Detectors	1998	VOA GC
GC09: Hewlett-Packard 5890 Series II with FID Detector	1999	VOA GC/MS Screen
GC10: Hewlett-Packard 5890A with FID/TCD Detectors	1999	VOA GC
GC11: Hewlett-Packard 5890 Series II+ with FID Detector (Combined with MS01)	1999	SVOA
GC12: Hewlett-Packard 5890 Series II+ with FID Detector (Combined with MS02)	2004	SVOA
GC13: Agilent 6890A Combined with Sievers 355 (SCD 2)	2001	VOA GC
GC14: Agilent 6890N with NPD/FID Detectors *Agilent 7683B Autosampler*	2005	SVOA
GC15: Agilent 6890N with NPD/FID Detectors *Agilent 7683 Autosampler*	2005	SVOA
GC16: Agilent 6890N with PFPD Detector and *OI Detector Controller* *Agilent 7683 Autosampler*	2005	SVOA
GC19: Hewlett-Packard 5890 with FID Detector	2007	VOA GC
GC20: Agilent 7890A with FID/TCD Detectors	2008	VOA GC
GC21:Hewlett-Packard 5890 Series II with ECD/FID Detectors	2009	SVOA
GC22: Agilent 7890A Combined with Agilent 355 (SCD 3)	2009	VOA GC
GC23: Hewlett-Packard 6890+ with ECD Detector (Combined with MS14)	2007	SVOA
GC24: Hewlett-Packard 5890 Series II (Combined with MS04)	2011	VOA GC
GC25: Hewlett-Packard 5890 Series II (Combined with MS12)	2006	SVOA
GC26: Agilent 7890A (Combined with MS19)	2011	VOA GC/MS
GC27: Agilent 7890A (Combined with MS20)	2011	VOA GC/MS

Equipment Description - GC/MS Systems	Purchased / Acquired	Location
MS01: HP 5890 Series II+ with FID Detector (GC11) & HP 5971A MSD *Hewlett-Packard 7673 Autosampler*	1991	SVOA
MS02: HP 5890 Series II+ with FID Detector (GC12) & HP 5972 MSD *Hewlett-Packard 7673 Autosampler*	1994	SVOA
MS04: HP 5890 Series II (GC24) & HP 5970 MSD	2004	VOA GC
MS05: Agilent 6890+/5973N MSD *Perkin Elmer TurboMatrix ATD-50 Thermal Desorber*	1999	VOA GC/MS
MS07: HP 6890A/ Agilent 5973N MSD	2001	SVOA
MS08: Agilent 6890N/5973inert MSD Tekmar AUTOCAN Autosampler	2004	VOA GC/MS
MS09: Agilent 6890N/5973inert MSD Tekmar AUTOCAN Autosampler	2005	VOA GC/MS
MS10: HP 6890A/5973 MSD	2006	SVOA
MS11: HP 5890 Series II/5972 MSD	2006	SVOA
MS12: HP 5890 Series II (GC25)/5971 MSD HP 7673 Autosampler	2006	SVOA
MS13: Agilent 6890N/5975B inert MSD Tekmar AUTOCAN Autosampler	2006	VOA GC/MS
MS14: HP 6890+ with ECD Detector (GC23) & HP 5973 MSD HP 6890 Injector	2007	SVOA
MS15: HP 5890 Series II/5972 MSD HP 7673 Autosampler	2007	SVOA
MS16: Agilent 6890N/5975C inert MSD Tekmar AUTOCAN Autosampler	2007	VOA GC/MS
MS17: Shimadzu GCMS QP-2010 Plus	2008	VOA GC/MS
MS18: Agilent 7890A /5975C inert XL MSD Markes Series 2 Unity Thermal Desorber Markes Series 2 Ultra TD Autosampler	2010	VOA GC/MS
MS19: Agilent 7890A (GC26) & 5975C inert XL MSD Tekmar AUTOCAN Autosampler	2011	VOA GC/MS
MS20: Agilent 7890A (GC27) & /5975C inert XL MSD Markes Series 2 Unity Thermal Desorber Markes Series 2 Ultra TD Autosampler	2011	VOA GC/MS
MS21: Agilent 7890A (GC28) & 5975C inert XL MSD Tekmar AUTOCAN Autosampler	2012	VOA GC/MS
MS22: Agilent 7890B (GC29) & 5977A MSD Markes CIA Advantage Autosampler	2015	VOA GC/MS

Liquid Chromatography	Purchased / Acquired	Location
LC03: Agilent Infinity LC 1220 (Combined with LCMS01)	2011	SVOA
LCMS01: Agilent 6120 Quadrupole MS (Combined with LC03)	2011	SVOA
Ion Chromatography	**Purchased / Acquired**	**Location**
IC03: Dionex ICS 2000 with Self-regenerating suppressor AS40 Autosampler	2008	GENCHEM
Spectrophotometer	**Purchased / Acquired**	**Location**
SPM01: Spectronic Instrument 20+ from SC	2001	GENCHEM
pH and Specific Ion Meters	**Purchased / Acquired**	**Location**
pH01: Thermo Orion 920 Selective Ion Meter	2001	GENCHEM
pH02: Orion 720A	1992	GENCHEM
Miscellaneous Equipment	**Purchased / Acquired**	**Location**
US Filter Water Purification System	2006	Main Lab
US Filter Water Purification System	2008	Extraction facility

Note: Purchase / Acquired year may represent when instrument was first maintained by ALS Environmental-Simi Valley or other in-network ALS Laboratory and does not reflect age of instrument.

Air sampling containers / Flow Controllers / Critical Orifices

Six-liter Summa passivated stainless steel canisters

- 1049 Ambient
- 1234 Source
- 191 Standard

Six-liter Silonite passivated stainless steel canisters

- 791 Ambient
- 334 Source

Three-liter Silco passivated stainless steel canisters (71)

One-liter Summa passivated stainless steel canisters (1112)

One-liter Silonite passivated stainless steel canisters (189)

400-millilter mini passivated stainless steel canisters (18)

Low volume flow controllers for time integrated sampling

- 863 Ambient
- 125 Source

Low-flow flow controllers for multi-day sampling (61)

Mini-canister flow controllers for time integrated sampling (16)

Critical orifices (2102)

Critical orifices – Sulfur (171)

Automated Summa Canister Conditioning Units

- Twenty-four position, microprocessor controlled conditioners with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (2)
- Ten position, microprocessor controlled conditioners with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (2)
- Fourteen position, microprocessor controlled conditioners with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (1)
- Sixteen position, microprocessor controlled conditioner with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (2)
- Six position, microprocessor controlled conditioner with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (1)

APPENDIX F – Containers, Preservation and Holding Times

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Maximum Holding Time
Solid / Water Sample Analysis				
Bromide (EPA 9056)	S,W	P, FP, G	Cool, 4˚C	28 Days
Chloride (EPA 9056)	S,W	P, FP, G	None Required	28 Days
Fluoride (9056)	S,W	P	Cool, 4˚C	28 days
Hydrogen Ion - pH (EPA 9040B/9045C)	S,W	P, FP, G	None Required	Analyze immediately
Nitrate, Nitrite (EPA 9056)	S,W	P, FP, G	Cool, 4˚C	48 hours
Orthophosphate (EPA 9056)	S	P,G	Cool, 4˚C	48 hours
Formaldehyde, Acetaldehyde (EPA 8315A Procedure 1 Modified)	S,W	Glass w/Teflon-Lined Lid	Cool, 4˚C	Aqueous – prep. - 72 hours, analysis – 30 days; Soil – prep. minimum, analysis – 30 days
Copper Corrosion (In-House Method)	Solid Wallboard	Ziploc Bag, G	None Required	-
H2S/Sulfur Emission (In-House Method)	Solid Wallboard	Ziploc Bag, G	None Required	-
Orthorhombic Cyclooctasulfur (In-House Method)	Solid Wallboard	Ziploc Bag, G	None Required	-

* W = Water or Aqueous solution; S = Soil or Sediment; P = Polyethylene, G = Glass, FP = fluoropolymer

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Maximum Holding Time	Sample Vol.[c]
Air Corrosivity	Air	Air Corrosivity Probes	Include 3 small dessicant bags (or equivalent) to each probe vial during shipment.	N/A[d]	3 Day Minimum Exposure
Amines (In-House Method)	Air	Treated Alumina Tubes	Sample Receipt-NA; Storage 4˚C±2˚C	30 days	100L
Ammonia (OSHA ID-188/ID-164)	Air	H_2SO_4 Treated Carbon Bead Tubes	Sample Receipt-NA; Storage 4˚C±2˚C	28 days	TWA: 24L STEL: 7.5L
BTU by ASTM D 3588 (SULFUR, ASTM D 5504; C1-C6+, EPA TO-3M; FIXED GASES, 3C)	Gaseous Fuels	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	<u>Sulfur</u> Bag – 24 hours Canister – 7 days[b] Bottle Vac[a] – 7 days[b] <u>C1-C6+</u> Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b] <u>3C</u> Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Carboxylic Acids (In-House Method)	Air	Treated Silica Gel Tubes	Sample Receipt-NA Storage 4˚C±2˚C	30 days until extraction; 14 days for analysis	100L
Total Gaseous Non-methane Organics (TGNMO) (EPA 25C)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Fixed Gases (EPA 3C & ASTM D 1946)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Helium & Hydrogen (EPA 3C Modified)	Air	Summa Canister Bottle Vac	N/A	Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Maximum Holding Time	Sample Vol.[c]
Argon (EPA 3C Modified)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours[b] Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Methane, Ethane, Ethene, Propane, Propene (RSK 175)	Aqueous	Glass w/Teflon- Lined Lid	No Headspace HCl to pH<2 4˚C±2˚C	14 days when preserved	(3) 40mL Vials
Carbon Dioxide (RSK 175)	Aqueous	Glass w/Teflon Lined Lid	No Headspace neutral pH (5-8) 4˚C±2˚C	N/A[d]	(3) 40mL Vials
Sulfur Compounds (In-House Method)	Aqueous	Glass w/Teflon Lined Lid	No Headspace; pH>4; 4˚C±2˚C	Following pH adjustment – 24 hours	(2) 40mL Vials
Sulfur Compounds (ASTM D 5504; SCAQMD 307-91; Modified SCAQMD 307-91)	Air	Tedlar Bag Fused Silica Lined Stainless Steel Canister Bottle Vac	No direct sunlight	Bag – 24 hours Canister – 7 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
C_1-C_6+ (EPA TO-3 Modified)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Methanol, Ethanol, Isopropyl alcohol, Freon, and Methylene Chloride (EPA TO-3 Modified)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Total Petroleum Hydrocarbons (TPHG) (EPA TO-3 Modified)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Pesticides and Polychlorinated Biphenyls (PCBs) (EPA TO-4A & TO-10A)	Air	Glass PUF Cartridge; TO-4A (High Volume); TO-10A (Low Volume)	Sample Receipt, <4˚C; Store sample and extract at <4˚C	7 days until extraction; extract – 40 days	2 m^3

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Maximum Holding Time	Sample Vol.[c]
Formaldehyde & Other Carbonyl Compounds (EPA TO-11A)	Air	DNPH-Coated Silica Gel Cartridge w/ Polypropylene Cap; SKC UME[x] and Bacharach GMD 570 Passive Monitors (formaldehyde only)	Sample Receipt, 4˚C±2˚C; Laboratory Preservation, 4˚C±2	14 days until extraction; 30 days for analysis	100 – 150L
Polycyclic Aromatic Hydrocarbons (PAHs) (EPA TO-13A)	Air	Polyurethane Foam (PUF) plugs, XAD Tube, PUF / XAD-2	Sample Receipt, <4˚C; Laboratory Preservation, <4˚C	7 days until extraction; 40 days after	130 – 400 m³
Volatile Organic Compounds (EPA TO-14A & TO-15)	Air	Tedlar Bag, Summa Canister (1L, 6L) Bottle Vac	N/A	Bag – 72 hours Canister – 30 days Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters 1.0L/6.0 Bottle Vacs 1.0L
Volatile Organic Compounds (EPA TO-17)	Air	Sorbent Tubes w/Swagelock Caps & PTFE Ferrules	<4˚C; organic solvent free environment; Laboratory Storage, 4˚C±2˚C	30 days	1-4L
Air-Phase Petroleum Hydrocarbons (MADEP APH)	Air	Summa Canister Bottle Vac	N/A	28 days Bottle Vac[a] – 30days[b]	Canisters 1.0L/6.0 Bottle Vacs 1.0L
Halogenated Volatile Organic Compounds (CARB 422)	Air	Tedlar Ba Summa Canister (1L, 6L) Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters 1.0L/6.0L Bottle Vacs 1.0L
Organic Vapors / NAPHTHAS (Diesel; etc.) (NIOSH 1550 / OSHA 7)	Air	Charcoal Tube; 3M 3500 or 3520 Badge; Silica Gel Tube w/ plastic caps	Sample Receipt-NA; Storage 4˚C±2˚C	14 days	Various
Sulfur Hexafluoride (NIOSH 6602 Modified)	Air	Tedlar Bag Summa Canister (1L, 6L)	N/A	Bag[b] – 72 hours Canister[a] – 30 days[b]	Bags 500mL Canisters 1.0L/6.0L
Siloxanes (In-House Method)	Air	SPE Cartidges Tedlar Bags	N/A	14 days until extraction; Tedlar Bags – transfer onto sorbent tube within 72 hours. 30 days for analysis	30L Cartridges Bags 500ml

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Holding Time	Sample Vol.[c]
Methanol, Acetaldehyde, Methyl Ethyl Ketone, Propionaldehyde (NCASI – DI/MeOH 94.03 /NCASI – DI/HAPS 99.01)	Aqueous - Effluent	Glass w/Teflon Lined Lid	No Headspace; 4˚C±2˚C; HCl to pH 2-3 (Effluent only)	30 days	(1) 40mL Vial
Reduced Sulfur Compounds (NCASI Method RSC-02.02)	Aqueous	40ml amber, borosilicate glass vials with Teflon faced silicone backed caps.	MeSH, DMS, and DMTS (RSCs non-H2S) addition of ascorbic acid and pH adjustment to <2.5 with 1:2 phosphoric acid solution upon collection. Laboratory Preservation, 4˚C±2	14 days	(2) 40ml VOA Vials
Total Sulfide (NCASI Method RSC-02.02)	Aqueous	40ml amber, borosilicate glass vials with Teflon faced silicone backed caps.	Addition of Zinc acetate solution and pH adjustment to >10 with 1 N NaOH solution upon collection. Laboratory Preservation, 4˚C±2	14 days	(2) 40ml VOA Vials
Hydrofluoric Acid (In-House Method)	Air	Radiello Samplers	Laboratory Preservation, 4˚C±2	4 months	15 minutes to 14 days exposure (dependent on sampling environment)
Hydrogen Sulfide (In-House Method)	Air	Radiello Samplers	N/A	6 months	1 hour to 15 days exposure (dependent on sampling environment)
Nitrogen Dioxide (In-House Method)	Air	Radiello samplers	Laboratory Preservation, store in dark at 4˚C±2	4 months	7 to 15 days exposure (dependent on sampling environment)

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Holding Time	Sample Vol.[c]
Ozone (In-House Method)	Air	Radiello Samplers	Protect from light	7 days	24 hours to 14 days exposure (dependent on sampling environment)
Sulfur Dioxide (In-House Method)	Air	Radiello Samplers	Laboratory Preservation, store in dark at 4˚C±2	4 months	7 to 15 days exposure (dependent on sampling environment)

Footnotes:

a.	Some methods do not specify the utilization of canisters; therefore, there is no required hold time and this will be noted in the case narrative.
b.	Laboratory recommended hold time; therefore, samples analyzed outside this hold time will be noted in the case narrative accordingly.
c.	Sample volumes are the minimum, which should be received by the laboratory; however, canister volumes should match the canister size utilized.
d.	There is no holding time requirement available and laboratory studies are not available indicating the validity of data prior to or following a specified length of time. Therefore, no holding time notation or qualifier will be adhered to results.

APPENDIX G – Standard Operating Procedures

Corporate SOP Titles	SOP ID
Laboratory Ethics and Data Integrity	CE-GEN001
(Proprietary – Client Specific)	CE-GEN002
Records Management Policy	CE-GEN003
Preventive Action	CE-GEN004
Document Control	CE-GEN005
Data Recall	CE-GEN006
Procurement and Control of Laboratory Services and Supplies	CE-GEN007
Method Development	CE-GEN008
Establishing Standard Operating Procedures	CE-GEN009
Handling Customer Feedback	CE-GEN010
Assigning a TSR to a Project	CE-GEN011
Policy for the use of Accreditation Organization Names, Symbols, and Logos	CE-GEN012
(Proprietary – Client Specific)	CE-GEN013
Internal Audits	CE-QA001
Manual Integration Policy	CE-QA002
Training Policy	CE-QA003
Qualification of Subcontract Laboratories	CE-QA004
Laboratory Management Review	CE-QA005
Proficiency Testing	CE-QA006
Making Entries onto Analytical Records	CE-QA007
Nonconformance and Corrective Action	CE-QA008
Control Limits	CE-QA009
Estimation of Uncertainty of Analytical Measurements	CE-QA010
Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	CE-QA011
Quality of Reagents and Standards	CE-QA012

Local Administrative SOP Titles	SOP Code
Data and Record Archiving	ADM-ARC
Batches and Sequences	ADM-BATCH_SEQ
Handling Consumable Materials	ADM-CONSUM
Electronic Data Backup, Archiving, and Restoration	ADM-DATA_BU
Data Review and Reporting	ADM-DATA_REV
Glassware Cleaning	ADM-GLASS
Analytical Instrument Acquisition, Reassignment, Maintenance and Documentation	ADM-INSTRUM
Laboratory Storage, Analysis, and Tracking	ADM-LabSAT
Media Request Fulfillment	ADM-Media_Req
Project Management	ADM-PMgmt
Software and Data Quality Assurance	ADM-SftwreQA
Significant Figures	ADM-SIG_FIG
Calibration and Use of Laboratory Support Equipment	ADM-SupEQ
Waste Disposal	ADM-WASTE
Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters	SMO-Can_Cert
Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters	SMO-Can_Press
Flow Controllers and Critical Orifices	SMO-Flow_Cntrl
Sample Receiving, Acceptance and Log-In	SMO-SMPL_REC

Semi-Volatile SOP Titles	SOP Code
Determination of Formaldehyde and Other Carbonyl Compounds in Ambient Air Using Adsorbent Cartridge Followed by High Performance Liquid Chromatography (HPLC) EPA Compendium Method T0-11A	SVO-11A
Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS)	SVO-13A
Determination of Volatile Amines in Ambient Air Using Gas Chromatography Equipped with a Nitrogen Phosphorus Detector (NPD)	SVO-AMINES
Determination of Carboxylic Acids in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS)	SVO-CACIDS
Analysis of Halogenated Volatile Organic Compounds in Emissions from Stationary Sources using GC/ECD in Accordance with a Modification of CARB Method 422	SVO-CARB422
NCASI Method RSC-02.02 Reduced Sulfur Compounds by Direct Injection GC/PFPD	SVO-NCASI_RSC
Determination of Methanol, Acetaldehyde, Methyl Ethyl Ketone, and Propionaldehyde in Pulp and Paper Process Liquids by GC/FID	SVO-NCASI_MeOH
Preparation and Analysis of 2-Butoxyethanol on Coconut Shell Charcoal Tubes and Analyzed using GC/FID	SVO-NIOSH1403
Determination of Organic Vapors Using GC/FID in Accordance with OSHA Method 07	SVO-OSHA_07
Determination of P-9290 Target Compounds from a Chamber and Specific P-9290 Quality Control Parameters	SVO-P9290
Preparation and Analysis of Orthorhombic Cyclooctasulfur by Gas Chromatography/Electron Capture Detector (GC/ECD)	SVO-S8_ECD
Analysis of Sulfur Hexafluoride in Accordance with a Modification of NIOSH 6602	SVO-SF6
Determination of Siloxanes in Biogas using Gas Chromatography/Mass Spectrometry (GC/MS)	SVO-SILOXANES
Determination of Pesticides and Polychlorinated Biphenyls (PCBs) in Ambient Air by GC/ECD per EPA Compendium Methods TO-4 and TO-10A	SVO-TO4A
Sample and Media Preparation per EPA Compendium Method TO-13A	SVP-TO13A
Sample Extraction and Preparation of Pesticide and PCB Samples According to EPA Compendium Methods TO-4A and TO-10A	SVP-TO4A

Volatile SOP Titles	SOP Code
Analysis of Air Corrosivity by Checkmate Meter	VOA-AIRCORR
Analysis of Argon Using Gas Chromatography with Thermal Conductivity Detection (TCD)	VOA-ARGON
Calculating Heat Value, Compressibility Factor, and Relative Density of Gaseous Fuels in Accordance with ASTM D 3588	VOA-BTU
Samples Preparation in Glass Chambers	VOA-CHAMBER
Dissolved Gas Analysis in Aqueous Samples Using a GC Headspace Equilibration Technique	VOA-DISGAS
Sample Preparation of Drywall for Sulfur Analysis and the Determination of Copper Corrosion	VOA-DRYWALL
Determination of Total Gaseous Nonmethane Organic (TGNMO) Emissions as Carbon in Landfill Gases in Accordance with EPA Method 25C	VOA-EPA25C
Determination of Methane, Carbon Monoxide, Carbon Dioxide, and Total Gaseous Nonmethane Organic (TGNMO) Emissions as Carbon in Landfill Gases According to Modified EPA Method 25C	VOA-EPA25CM
Determination of Hydrogen, Carbon Monoxide, Carbon Dioxide, Nitrogen, Methane, and Oxygen using Gas Chromatography with Thermal Conductivity Detection (TCD) in Accordance with EPA 3C or ASTM D 1946	VOA-EPA3C
Analysis of Hydrogen and Helium using Gas Chromatography with Thermal Conductivity Detection (TCD)	VOA-HHe
Analysis of Sulfur Compounds in a Gaseous Matrix by Gas Chromatography with Sulfur Chemiluminescence Detection per ASTM D 5504 and Modified SCAQMD Method 307	VOA-S307M_SCD
Analysis of Sulfur Compounds in Liquid Samples by Gas Chromatography with Sulfur Chemiluminescence Detection	VOA-SH_2O_SCD
Analysis of C1-C6+ using Gas Chromatography with Flame Ionization Detection (FID) in Accordance with a Modification of EPA Compendium Method TO-3	VOA-TO3C1C6
Analysis of Various Compounds using Gas Chromatography with Flame Ionization Detection (FID) in Accordance with a Modification of EPA Compendium Method TO-3	VOA-TO3MeOH
Analysis of Total Petroleum Hydrocarbons as Gasoline in Air by Gas Chromatography with Flame Ionization Detection	VOA-TPHG_TO3
Determination of Air-Phase Petroleum Hydrocarbons by Gas Chromatography/Mass Spectrometry (GC/MS)	VOA-MAPH
Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)	VOA-TO15
Determination of Volatile Organic Compounds in Ambient Air Using Active or Passive Sampling Onto Sorbent Tubes	VOA-TO17

General Chemistry (WET) SOP Titles	SOP Code
Determination of Inorganic Anions by Ion Chromatography	WET-Anions_IC
Colorimetric Determination of Hydrogen Sulfide (H_2S) in Air	WET-H_2SAir
Analysis of Hydrofluoric (HF) Acid in Air by Ion Selective Electrode	WET-HFAir
Ammonia in Air by Ion Selective Electrode	WET-NH_3Air
Colorimetric Determination of Nitrogen Dioxide (NO_2) in Air	WET-NO_2Air
Colorimetric Determination of Ozone (O_3) in Air	WET-O_3Air
pH Electrometric Measurement for Liquids by Ion Selective Electrodes	WET-pHL
pH Electrometric Measurement for Solids by Ion Selective Electrodes	WET-pHS

APPENDIX H – Data Qualifiers

CODE	CATEGORY	DESCRIPTION
BC	AIHA	Reported results are not blank corrected.
BH	AIHA	Results indicate breakthrough; back section of tube greater than front section.
BT	AIHA	Results indicated possible breakthrough; back section ≥10% front section.
DE	AIHA	Reported results are corrected for desorption efficiency.
RA	AIHA	Result not available.
G	GENERAL	Improper container.
G1	GENERAL	Unpreserved or improperly preserved sample.
X	GENERAL	See case narrative.
H1	HOLD TIME	Sample analysis performed past holding time. See case narrative.
H2	HOLD TIME	Initial analysis within holding time. Reanalysis for the required dilution was past holding time.
H3	HOLD TIME	Sample was received and analyzed past holding time.
H4	HOLD TIME	Sample was extracted past required extraction holding time, but analyzed within analysis holding time. See case narrative.
i	MATRIX	The MDL/MRL has been elevated due to matrix interference.
M	MATRIX	Matrix interference; results may be biased (high/low).
M1	MATRIX	Matrix interference due to coelution with a non-target compound. (TO-15 only)
Q	PETROLEUM	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of lighter/heavier molecular weight constituents than the calibration standard.
Y	PETROLEUM	The chromatogram resembles a petroleum product but does not match the calibration standard.
Z	PETROLEUM	The chromatogram does not resemble a petroleum product.
#	QC	The control limit criterion is not applicable. See case narrative.
*	QC	The result is an outlier. See case narrative.
B	QC	Analyte detected in both the sample and associated method blank.
I	QC	Internal standard not within the specified limits. See case narrative.
L	QC	Laboratory control sample recovery outside the specified limits; results may be biased (high/low).
N	QC	The matrix spike sample recovery is not within control limits. See case narrative.
R	QC	Duplicate precision not met.

CODE	CATEGORY	DESCRIPTION
R1	QC	Duplicate precision not within the specified limits; however, the results are below the MRL and considered estimated.
S	QC	Surrogate recovery not within specified limits.
V	QC	The continuing calibration verification standard was outside (biased high/low) the specified limits for this compound.
C	RESULT	Result identification confirmed.
CE	RESULT	Co-elution.
D	RESULT	The reported result is from a dilution.
E	RESULT	Estimated; concentration exceeded calibration range.
J	RESULT	The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
J1	RESULT	The analyte was positively identified below the method reporting limit prior to utilizing the dilution factor; the associated numerical value is considered estimated.
K	RESULT	Analyte was detected above the method reporting limit prior to normalization.
ND	RESULT	Compound was analyzed for, but not detected above the laboratory reporting/detection limit.
P	RESULT	The confirmation criterion was exceeded. The relative percent difference was greater than 40/25% between the two analytical results.
U	RESULT	Compound was analyzed for, but not detected (ND) at or above the MRL/MDL.
W	RESULT	Result quantified, but the corresponding peak was detected outside the generated retention time window.
UJ	RESULT	The analyte was not detected; however, the result is estimated due to discrepancies in meeting certain analyte-specific quality control criteria.
Ui	RESULT	The compound was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL; however, the MRL/MDL has been elevated due to matrix interference.
T	TIC	Analyte is a tentatively identified compound, result is estimated.

APPENDIX I – Master List of Controlled Documents

Controlled Documents*	Document Code
Health and Safety Manual	ADM-SAFETY
Quality Assurance Manual	ALSMV-QAM

*Refer to Appendix G for a list of the laboratory's controlled standard operating procedures.

QA Program Files	
Item	**Location / Name**
Approved Signatories List	QA Manual Appendix I
Approved Subcontract Laboratories	Q:\Approved Sub-Contract Labs\Subcontract Lab List
Control Limit\Chart Status	Q:\Control Charts\CntrlChrt(status1).xls
Job Descriptions	HR Department
Master List of Controlled Documents (Logbooks, SOPs, etc.)	Q:\Master List of Controlled Documents\Master List of Controlled Documents.xls
MDL,LOD,LOQ Status	Q:\MDL Status\MDL Status Table (EACH DEPT).xls
Personnel Resumes, Transcripts	HR and QA Departments
Simi Valley Certification Status	Q:\Certifications\Cert Status.xls
Simi Valley Data Quality Objectives	Q:\MDL_MRL\DQO Spreadsheet.xls
Technical Training Status	Q:\Training\TRAINING STATUS\TRAINING STATUS.xls

Approved Signatories	
Name	**Title**
Kelly Horiuchi, B.A.	Laboratory Director / Project Manager
Chaney Humphrey, B.S.	Quality Assurance Manager
Wade Henton, B.S.	Volatiles (GC) Technical Manager
Chris Parnell, B.S.	Operations Manager; Technical Manager (VOA GC/MS – Air)
Madeleine Dangazyan, B.S.	Semi-Volatiles/ Industrial Hygiene Technical Manager; Environmental Health & Safety Coordinator
Wida Ang, B.S., M.S.	Team Leader (Volatiles GC/MS – Air)
Sue Anderson, B.S.	Project Manager / Technical Manager (General Chemistry)
Samantha Henningsen, B.S.	Project Manager
Kathleen Aguilera, B.A.	Client Services Manager / Project Manager

APPENDIX J – Laboratory Accreditations

American Industrial Hygiene Association (AIHA)

Industrial Hygiene Laboratory Accreditation Program Laboratory
Laboratory # 101661
Approved Method(s):

- NIOSH 1450
- NIOSH 1457
- NIOSH 1500
- NIOSH 1501
- NIOSH 1550
- OSHA 07

State of Arizona, Department of Health Services

License No. AZ0694
Approved Method(s):

- EPA TO-15
- EPA 3C

Department of Defense, Environmental Laboratory Accreditation Program (DoD-ELAP)

Perry Johnson Laboratory Accreditation, Inc. Accreditation No. 65818
Approved Method(s):

- EPA TO-15
- RSK 175
- EPA 3C
- ASTM D 1946-90
- SOP VOA-EPA3C (EPA 3C Modified)
- SOP VOA-TPHG_TO3 (TPHG by Modified EPA TO-3)
- SOP VOA-TO3C1C6 (Hydrocarbons and ranges by Modified EPA TO-3)
- SOP VOA-TO15 (EPA TO-15 Modified)

State of Florida, Department of Health (NELAP-Secondary)

Laboratory ID No.: E871020
Approved Method(s):

- EPA TO-15
- EPA TO-17

State of Maine, Department of Health and Human Services

Certificate No.: 2014025
Approved Methods

- EPA TO-15
- MADEP APH

State of Minnesota, Department of Health, Environmental Laboratory Certification Program (NELAP-Secondary)
Laboratory ID: 006-999-456
Approved Method(s):
- EPA TO-15

State of New York, Department of Health (NELAP –Secondary)
Environmental Analyses/Air and Emissions
Laboratory ID No. 11221
Approved Method(s):
- EPA TO-13A
- EPA TO-15
- EPA TO-17

State of New Jersey, Department of Environmental Protection (NELAP-Secondary)
Laboratory ID: CA009
Approved Method(s):
- EPA TO-15
- EPA TO-13A

State of Oregon, Environmental Laboratory Accreditation Program (NELAP-Primary)
Laboratory ID: 4068
Approved Method(s):
- EPA TO-4A
- EPA TO-10A
- EPA TO13A
- EPA TO-15
- EPA TO-17
- MADEP APH

Commonwealth of Pennsylvania, Department of Environmental Protection Bureau of Laboratories
Registration Number: 68-03307

State of Texas, Texas Commission on Environmental Quality (NELAP-Secondary)
Certificate # T104704413-14-5
Approved Method(s):
- EPA TO-15

State of Utah, Department of Health, Environmental Laboratory Certification Program (NELAP-Secondary)
Certificate # CA016272014-4
Approved Method(s):
- EPA TO-15

State of Washington, Department of Ecology
Laboratory ID: C946
Approved Method(s):
- EPA TO-15
- EPA RSK-175

Note 1: This Quality Assurance Manual is revised annually with AIHA, DoD and NELAP-Primary Certificates, and the Scope of Accreditations/Parameters are revised annually (where necessary). During this interim period Certificates may expire and the Scope of Accreditations/Parameters may change; therefore, these may not be updated until the next revision.

Note 2: Current Certificates and Scope of Accreditations/Parameters are on file and displayed in the front hallway. Updated or Specific Certificates and Scope of Accreditations/Parameters are available upon request.

AIHA Laboratory Accreditation Programs, LLC

acknowledges that

ALS Environmental – Simi Valley
2655 Park Center Drive, Suite A, Simi Valley, CA 93065-6200
Laboratory ID: 101661

along with all premises from which key activities are performed, as listed above, has fulfilled the requirements of the AIHA Laboratory Accreditation Programs (AIHA-LAP), LLC accreditation to the ISO/IEC 17025:2005 international standard, *General Requirements for the Competence of Testing and Calibration Laboratories* in the following:

LABORATORY ACCREDITATION PROGRAMS

✓	**INDUSTRIAL HYGIENE**	Accreditation Expires: 11/01/2016
☐	**ENVIRONMENTAL LEAD**	Accreditation Expires:
☐	**ENVIRONMENTAL MICROBIOLOGY**	Accreditation Expires:
☐	**FOOD**	Accreditation Expires:
☐	**UNIQUE SCOPES**	Accreditation Expires:

Specific Field(s) of Testing (FoT)/Method(s) within each Accred
itation Program for which the above named laboratory maintains accreditation is outlined on the attached **Scope of Accreditation**. Continued accreditation is contingent upon successful on-going compliance with ISO/IEC 17025:2005 and AIHA-LAP, LLC requirements. This certificate is not valid without the attached **Scope of Accreditation**. Please review the AIHA-LAP, LLC website (www.aihaaccreditedlabs.org) for the most current Scope.

Gerald Schultz, CIH
Chairperson, Analytical Accreditation Board

Cheryl O. Morton
Managing Director, AIHA Laboratory Accreditation Programs, LLC

Revision 14: 03/26/2014

Date Issued: 09/30/2014

AIHA Laboratory Accreditation Programs, LLC
SCOPE OF ACCREDITATION

ALS Environmental – Simi Valley
2655 Park Center Drive Suite A, Simi Valley, CA 93065-6200

Laboratory ID: **101661**
Issue Date: 09/30/2014

The laboratory is approved for those specific field(s) of testing/methods listed in the table below. Clients are urged to verify the laboratory's current accreditation status for the particular field(s) of testing/Methods, since these can change due to proficiency status, suspension and/or withdrawal of accreditation.

Industrial Hygiene Laboratory Accreditation Program (IHLAP)

Initial Accreditation Date: 09/01/1994

IHLAP Scope Category	Field of Testing (FoT)	Technology sub-type/ Detector	Published Reference Method/Title of In-house Method	Method Description or Analyte *(for internal methods only)*
Chromatography Core	Gas Chromatography	GC/FID	NIOSH 1450	
			NIOSH 1457	
			NIOSH 1500	
			NIOSH 1501	
			NIOSH 1550	
			OSHA 07	
	Gas Chromatography (Diffusive Samplers)		OSHA 07	

A complete listing of currently accredited Industrial Hygiene laboratories is available on the AIHA-LAP, LLC website at: http://www.aihaaccreditedlabs.org

Effective: 03/12/2013
101661_Scope_IHLAP_2014_09_30.docx
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PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

ALS Environmental
2655 Park Center Drive, Suite A, Simi Valley, CA 93065

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the DoD Quality Systems Manual for Environmental Laboratories Version 4.2 10/26/2010 and is accredited is accordance with the:

United States Department of Defense
Environmental Laboratory Accreditation Program
(DoD-ELAP)

This accreditation demonstrates technical competence for the defined scope:
Environmental Testing
(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body's duty to observe and comply with the said rules.

For PJLA:

Tracy Szerszen
President/Operations Manager

Initial Accreditation Date:	*Issue Date:*	*Expiration Date:*
January 11, 2010	January 2, 2014	January 31, 2016

Accreditation No.:	*Certificate No.:*
65818	L14-2

Perry Johnson Laboratory
Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Aqueous	RSK 175	GC/FID	Methane
Aqueous	RSK 175	GC/FID	Ethane
Aqueous	RSK 175	GC/FID	Ethene
Aqueous	RSK 175	GC/TCD	Carbon Dioxide
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Hydrogen
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Oxygen
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Nitrogen
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Methane
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Carbon Dioxide
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Carbon Monoxide
Air	(ALS SOP) VOA-TPHG_TO3	GC/FID	Total Petroleum Hydrocarbons Gasoline (TPHG)
Air	(ALS SOP) VOA-TPHG_TO3	GC/FID	JP-4
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	C1 - C6+
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Ethane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Ethene
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Methane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	n-Butane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	n-Hexane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	n-Pentane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Propane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Propene
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Total Volatile Petroleum Hydrocarbons (TVPH) as Hexane
Air	EPA TO-15	GC/MS	1,1,1-Trichloroethane
Air	EPA TO-15	GC/MS	1,1,2,2-Tetrachloroethane
Air	EPA TO-15	GC/MS	1,1,2-Trichloroethane
Air	EPA TO-15	GC/MS	1,1-Dichloroethane
Air	EPA TO-15	GC/MS	1,1-Dichloroethene
Air	EPA TO-15	GC/MS	1,2,3-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,2,4-Trichlorobenzene

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 2 of 8*

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	1,2,4-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,2,Dibromo-3-Chloropropane
Air	EPA TO-15	GC/MS	1,2-Dibromoethane
Air	EPA TO-15	GC/MS	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
Air	EPA TO-15	GC/MS	1,2-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,2-Dichloroethane
Air	EPA TO-15	GC/MS	1,2-Dichloropropane
Air	EPA TO-15	GC/MS	1,3,5-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,3-Butadiene
Air	EPA TO-15	GC/MS	1,3-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,4-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,4-Dioxane
Air	EPA TO-15	GC/MS	1-Butanol
Air	EPA TO-15	GC/MS	2-Butanone (MEK)
Air	EPA TO-15	GC/MS	2-Ethyltoluene
Air	EPA TO-15	GC/MS	2-Hexanone
Air	EPA TO-15	GC/MS	3-Ethyltoluene
Air	EPA TO-15	GC/MS	4-Ethyltoluene
Air	EPA TO-15	GC/MS	4-Methyl-2-Pentanone
Air	EPA TO-15	GC/MS	Acetone
Air	EPA TO-15	GC/MS	Acetonitrile
Air	EPA TO-15	GC/MS	Acrolein
Air	EPA TO-15	GC/MS	Acrylonitrile
Air	EPA TO-15	GC/MS	Allyl Chloride
Air	EPA TO-15	GC/MS	alpha-Methylstyrene
Air	EPA TO-15	GC/MS	alpha-Pinene
Air	EPA TO-15	GC/MS	Benzene
Air	EPA TO-15	GC/MS	Benzyl Chloride
Air	EPA TO-15	GC/MS	Bromodichloromethane
Air	EPA TO-15	GC/MS	Bromoform
Air	EPA TO-15	GC/MS	Bromomethane
Air	EPA TO-15	GC/MS	Carbon Disulfide
Air	EPA TO-15	GC/MS	Carbon Tetrachloride
Air	EPA TO-15	GC/MS	Chlorobenzene
Air	EPA TO-15	GC/MS	Chloroethane

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 3 of 8*

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	Chloroform
Air	EPA TO-15	GC/MS	Chloromethane
Air	EPA TO-15	GC/MS	cis-1,2-Dichloroethene
Air	EPA TO-15	GC/MS	cis-1,3-Dichloropropene
Air	EPA TO-15	GC/MS	Cumene
Air	EPA TO-15	GC/MS	Cyclohexane
Air	EPA TO-15	GC/MS	Cyclohexanone
Air	EPA TO-15	GC/MS	Dibromochloromethane
Air	EPA TO-15	GC/MS	Dichlorodifluoromethane (CFC 12)
Air	EPA TO-15	GC/MS	Diisopropyl Ether
Air	EPA TO-15	GC/MS	d-Limonene
Air	EPA TO-15	GC/MS	Ethanol
Air	EPA TO-15	GC/MS	Ethyl Acetate
Air	EPA TO-15	GC/MS	Ethyl tert-Butyl Ether
Air	EPA TO-15	GC/MS	Ethylbenzene
Air	EPA TO-15	GC/MS	Hexachlorobutadiene
Air	EPA TO-15	GC/MS	Isooctane
Air	EPA TO-15	GC/MS	Isopropyl acetate
Air	EPA TO-15	GC/MS	Isopropyl Alcohol
Air	EPA TO-15	GC/MS	m-&, p-Xylenes
Air	EPA TO-15	GC/MS	Methyl Methacrylate
Air	EPA TO-15	GC/MS	Methyl tert-Butyl Ether
Air	EPA TO-15	GC/MS	Methylene Chloride
Air	EPA TO-15	GC/MS	Naphthalene
Air	EPA TO-15	GC/MS	n-Butyl Acetate
Air	EPA TO-15	GC/MS	n-Butylbenzene
Air	EPA TO-15	GC/MS	n-Decane
Air	EPA TO-15	GC/MS	n-Dodecane
Air	EPA TO-15	GC/MS	n-Heptane
Air	EPA TO-15	GC/MS	n-Hexane
Air	EPA TO-15	GC/MS	n-Nonane

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 4 of 8*

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	n-Octane
Air	EPA TO-15	GC/MS	n-Propylbenzene
Air	EPA TO-15	GC/MS	n-Undecane
Air	EPA TO-15	GC/MS	o-Xylene
Air	EPA TO-15	GC/MS	p-Isopropyltoluene
Air	EPA TO-15	GC/MS	Propene
Air	EPA TO-15	GC/MS	sec-Butylbenzene
Air	EPA TO-15	GC/MS	Styrene
Air	EPA TO-15	GC/MS	tert-Amyl Methyl Ether
Air	EPA TO-15	GC/MS	tert-Butanol
Air	EPA TO-15	GC/MS	tert-Butylbenzene
Air	EPA TO-15	GC/MS	Tetrachloroethene
Air	EPA TO-15	GC/MS	Tetrahydrofuran
Air	EPA TO-15	GC/MS	Toluene
Air	EPA TO-15	GC/MS	trans-1,2-Dichloroethene
Air	EPA TO-15	GC/MS	trans-1,3-Dichloropropene
Air	EPA TO-15	GC/MS	Trichloroethene
Air	EPA TO-15	GC/MS	Trichlorofluoromethane
Air	EPA TO-15	GC/MS	Trichlorotrifluoroethane
Air	EPA TO-15	GC/MS	Vinyl Acetate
Air	EPA TO-15	GC/MS	Vinyl Chloride
Air	ASTM D 1946-90	GC/TCD	Hydrogen
Air	ASTM D 1946-90	GC/TCD	Oxygen
Air	ASTM D 1946-90	GC/TCD	Nitrogen
Air	ASTM D 1946-90	GC/TCD	Methane
Air	ASTM D 1946-90	GC/TCD	Carbon Dioxide
Air	ASTM D 1946-90	GC/TCD	Carbon Monoxide
Air	EPA 3C	GC/TCD	Oxygen
Air	EPA 3C	GC/TCD	Nitrogen
Air	EPA 3C	GC/TCD	Methane
Air	EPA 3C	GC/TCD	Carbon Dioxide
Air	(ALS SOP) VOA-TO15	GC/MS	1,1,1-Trichloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,1,2,2-Tetrachloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,1,2-Trichloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,1-Dichloroethane

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 5 of 8*

Certificate of Accreditation: Supplement

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ALS Environmental

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	(ALS SOP) VOA-TO15	GC/MS	1,1-Dichloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2,3-Trimethylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2,4-Trichlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2,4-Trimethylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dibromo-3-Chloropropane
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dibromoethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dichlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dichloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dichloropropane
Air	(ALS SOP) VOA-TO15	GC/MS	1,3,5-Trimethylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,3-Butadiene
Air	(ALS SOP) VOA-TO15	GC/MS	1,3-Dichlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,4-Dichlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,4-Dioxane
Air	(ALS SOP) VOA-TO15	GC/MS	1-Butanol
Air	(ALS SOP) VOA-TO15	GC/MS	2-Butanone (MEK)
Air	(ALS SOP) VOA-TO15	GC/MS	2-Ethyltoluene
Air	(ALS SOP) VOA-TO15	GC/MS	2-Hexanone
Air	(ALS SOP) VOA-TO15	GC/MS	3-Ethyltoluene
Air	(ALS SOP) VOA-TO15	GC/MS	4-Ethyltoluene
Air	(ALS SOP) VOA-TO15	GC/MS	4-Methyl-2-Pentanone
Air	(ALS SOP) VOA-TO15	GC/MS	Acetone
Air	(ALS SOP) VOA-TO15	GC/MS	Acetonitrile
Air	(ALS SOP) VOA-TO15	GC/MS	Acrolein
Air	(ALS SOP) VOA-TO15	GC/MS	Acrylonitrile
Air	(ALS SOP) VOA-TO15	GC/MS	Allyl Chloride
Air	(ALS SOP) VOA-TO15	GC/MS	alpha-Methylstyrene
Air	(ALS SOP) VOA-TO15	GC/MS	alpha-Pinene
Air	(ALS SOP) VOA-TO15	GC/MS	Benzene
Air	(ALS SOP) VOA-TO15	GC/MS	Benzyl Chloride
Air	(ALS SOP) VOA-TO15	GC/MS	Bromodichloromethane
Air	(ALS SOP) VOA-TO15	GC/MS	Bromoform
Air	(ALS SOP) VOA-TO15	GC/MS	Bromomethane

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 6 of 8*

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	(ALS SOP) VOA-TO15	GC/MS	Carbon Disulfide
Air	(ALS SOP) VOA-TO15	GC/MS	Carbon Tetrachloride
Air	(ALS SOP) VOA-TO15	GC/MS	Chlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	Chloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	Chloroform
Air	(ALS SOP) VOA-TO15	GC/MS	Chloromethane
Air	(ALS SOP) VOA-TO15	GC/MS	cis-1,2-Dichloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	cis-1,3-Dichloropropene
Air	(ALS SOP) VOA-TO15	GC/MS	Cumene
Air	(ALS SOP) VOA-TO15	GC/MS	Cyclohexane
Air	(ALS SOP) VOA-TO15	GC/MS	Cyclohexanone
Air	(ALS SOP) VOA-TO15	GC/MS	Dibromochloromethane
Air	(ALS SOP) VOA-TO15	GC/MS	Dichlorodifluoromethane (CFC 12)
Air	(ALS SOP) VOA-TO15	GC/MS	Diisopropyl Ether
Air	(ALS SOP) VOA-TO15	GC/MS	d-Limonene
Air	(ALS SOP) VOA-TO15	GC/MS	Ethanol
Air	(ALS SOP) VOA-TO15	GC/MS	Ethyl Acetate
Air	(ALS SOP) VOA-TO15	GC/MS	Ethyl tert-Butyl Ether
Air	(ALS SOP) VOA-TO15	GC/MS	Ethylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	Hexachlorobutadiene
Air	(ALS SOP) VOA-TO15	GC/MS	Isooctane
Air	(ALS SOP) VOA-TO15	GC/MS	Isopropyl acetate
Air	(ALS SOP) VOA-TO15	GC/MS	Isopropyl Alcohol
Air	(ALS SOP) VOA-TO15	GC/MS	m-&,p-Xylenes
Air	(ALS SOP) VOA-TO15	GC/MS	Methyl Methacrylate
Air	(ALS SOP) VOA-TO15	GC/MS	Methyl tert-Butyl Ether
Air	(ALS SOP) VOA-TO15	GC/MS	Methylene Chloride
Air	(ALS SOP) VOA-TO15	GC/MS	Naphthalene
Air	(ALS SOP) VOA-TO15	GC/MS	n-Butyl Acetate
Air	(ALS SOP) VOA-TO15	GC/MS	n-Butylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	n-Decane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Dodecane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Heptane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Hexane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Nonane

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 7 of 8*

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	(ALS SOP) VOA-TO15	GC/MS	n-Octane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Propylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	n-Undecane
Air	(ALS SOP) VOA-TO15	GC/MS	o-Xylene
Air	(ALS SOP) VOA-TO15	GC/MS	p-Isopropyltoluene
Air	(ALS SOP) VOA-TO15	GC/MS	Propene
Air	(ALS SOP) VOA-TO15	GC/MS	sec-Butylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	Styrene
Air	(ALS SOP) VOA-TO15	GC/MS	tert-Amyl Methyl Ether
Air	(ALS SOP) VOA-TO15	GC/MS	t-Butanol
Air	(ALS SOP) VOA-TO15	GC/MS	tert-Butylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	Tetrachloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	Tetrahydrofuran
Air	(ALS SOP) VOA-TO15	GC/MS	Toluene
Air	(ALS SOP) VOA-TO15	GC/MS	trans-1,2-Dichloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	trans-1,3-Dichloropropene
Air	(ALS SOP) VOA-TO15	GC/MS	Trichloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	Trichlorofluoromethane
Air	(ALS SOP) VOA-TO15	GC/MS	Trichlorotrifluoroethane
Air	(ALS SOP) VOA-TO15	GC/MS	Vinyl Acetate
Air	(ALS SOP) VOA-TO15	GC/MS	Vinyl Chloride

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 8 of 8*

OREGON

Environmental Laboratory Accreditation Program

NELAP Recognized

ALS Environmental - Simi Valley

4068

2655 Park Center Drive, Suite A

Simi Valley,CA 93065

IS GRANTED APPROVAL BY ORELAP UNDER THE 2009 TNI STANDARDS, TO PERFORM ANALYSES ON ENVIRONMENTAL SAMPLES IN MATRICES AS LISTED BELOW :

Air	*Drinking Water*	*Non Potable Water*	*Solids and Chem. Waste*	*Tissue*
Chemistry		Chemistry		

AND AS RECORDED IN THE LIST OF APPROVED ANALYTES, METHODS, ANALYTICAL TECHNIQUES, AND FIELDS OF TESTING ISSUED CONCURRENTLY WITH THIS CERTIFICATE AND REVISED AS NECESSARY.

ACCREDITED STATUS DEPENDS ON SUCCESSFUL ONGOING PARTICIPATION IN THE PROGRAM AND CONTINUED COMPLIANCE WITH THE STANDARDS.

CUSTOMERS ARE URGED TO VERIFY THE LABORATORY'S CURRENT ACCREDITATION STATUS IN OREGON.

Gary K. Ward, MS
Oregon State Public Health Laboratory
ORELAP Administrator
3150 NW. 229th Ave, Suite 100
Hillsboro, OR 97124

ISSUE DATE: 02/16/2015
EXPIRATION DATE: 02/15/2016
Certificate No: 4068 - 001

Uncontrolled Copy

Oregon

Environmental Laboratory Accreditation Program

Department of Agriculture, Laboratory Division
Department of Environmental Quality, Laboratory Division
Oregon Health Authority, Public Health Division

NELAP Recognized

ORELAP Fields of Accreditation

ORELAP ID: 4068
EPA CODE: CA01627
Certificate: 4068 - 001

ALS Environmental - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley CA 93065

Issue Date: 02/16/2015 ***Expiration Date:*** 02/15/2016

As of 02/16/2015 ***this list supercedes all previous lists for this certificate number. Customers. Please verify the current accreditation standing with ORELAP.***

MATRIX : Air

Reference	Code	Description
EPA TO-10A (GC/ECD)	**10247504**	**Pesticides and PCBs with LV PUF by GC/ECD**

Analyte Code	*Analyte*
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
8880	Aroclor-1016 (PCB-1016)
8910	Aroclor-1260 (PCB-1260)
7115	beta-BHC (beta-Hexachlorocyclohexane)
7105	delta-BHC
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide
7810	Methoxychlor

EPA TO-13A	**10248405**	**Polycyclic Aromatic Hydrocarbons in Ambient Air by GC/MS**

Analyte Code	*Analyte*
5500	Acenaphthene
5505	Acenaphthylene
5555	Anthracene
5575	Benzo(a)anthracene
5580	Benzo(a)pyrene
5590	Benzo(g,h,i)perylene
5600	Benzo(k)fluoranthene
5585	Benzo[b]fluoranthene
5855	Chrysene
5895	Dibenz(a,h) anthracene
6265	Fluoranthene
6270	Fluorene
6315	Indeno(1,2,3-cd) pyrene

ORELAP Fields of Accreditation

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ALS Environmental - Simi Valley
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Simi Valley CA 93065

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Analyte Code	*Analyte*
5005	Naphthalene
6615	Phenanthrene
6665	Pyrene

EPA TO-15 **10248803** **VOCs collected in Canisters by GC/MS**

Analyte Code	*Analyte*
5160	1,1,1-Trichloroethane
5110	1,1,2,2-Tetrachloroethane
5195	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
5165	1,1,2-Trichloroethane
4630	1,1-Dichloroethane
4640	1,1-Dichloroethylene
5182	1,2,3-Trimethylbenzene
5155	1,2,4-Trichlorobenzene
5210	1,2,4-Trimethylbenzene
4570	1,2-Dibromo-3-chloropropane (DBCP)
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
4695	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane (Ethylene dichloride)
4655	1,2-Dichloropropane
5215	1,3,5-Trimethylbenzene
9318	1,3-Butadiene
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
4735	1,4-Dioxane (1,4- Diethyleneoxide)
4836	1-Propene
5220	2,2,4-Trimethylpentane
4410	2-Butanone (Methyl ethyl ketone, MEK)
4538	2-Ethyltoluene
4860	2-Hexanone
4531	3-Ethyltoluene
4542	4-Ethyltoluene
4910	4-Isopropyltoluene (p-Cymene)
4995	4-Methyl-2-pentanone (MIBK)
4315	Acetone
4320	Acetonitrile
4325	Acrolein (Propenal)
4340	Acrylonitrile
4355	Allyl chloride (3-Chloropropene)
4357	alpha-Methylstyrene
6698	alpha-Pinene
4375	Benzene
5635	Benzyl chloride
4395	Bromodichloromethane
4400	Bromoform
4450	Carbon disulfide
4455	Carbon tetrachloride
4475	Chlorobenzene
4575	Chlorodibromomethane
4485	Chloroethane (Ethyl chloride)
4505	Chloroform
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4555	Cyclohexane
4560	Cyclohexanone
4625	Dichlorodifluoromethane (Freon-12)
9375	Di-isopropylether (DIPE)
6208	d-Limonene
4750	Ethanol

ORELAP Fields of Accreditation

ORELAP ID: 4068
EPA CODE: CA01627
Certificate: 4068 - 001

ALS Environmental - Simi Valley

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Simi Valley CA 93065

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Analyte Code	*Analyte*
4755	Ethyl acetate
4765	Ethylbenzene
4770	Ethyl-t-butylether (ETBE) (2-Ethoxy-2-methylpropane)
4835	Hexachlorobutadiene
4890	Isopropyl acetate
4895	Isopropyl alcohol (2-Propanol, Isopropanol)
4900	Isopropylbenzene
5240	m+p-xylene
4950	Methyl bromide (Bromomethane)
4960	Methyl chloride (Chloromethane)
4990	Methyl methacrylate
5000	Methyl tert-butyl ether (MTBE)
4975	Methylene chloride (Dichloromethane)
5005	Naphthalene
4425	n-Butyl alcohol (1-Butanol, n-Butanol)
4415	n-Butyl-acetate
4435	n-Butylbenzene
5875	n-Decane
6235	n-Dodecane
4825	n-Heptane
4855	n-Hexane
5026	n-Nonane
5027	n-Octane
5090	n-Propylbenzene
6747	n-Undecane
5250	o-Xylene
4440	sec-Butylbenzene
5100	Styrene
4370	T-amylmethylether (TAME)
4420	tert-Butyl alcohol
4445	tert-Butylbenzene
5115	Tetrachloroethylene (Perchloroethylene)
5120	Tetrahydrofuran (THF)
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
5225	Vinyl acetate
5235	Vinyl chloride
5260	Xylene (total)

EPA TO-17 **10312206** **Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes**

Analyte Code	*Analyte*
5160	1,1,1-Trichloroethane
5110	1,1,2,2-Tetrachloroethane
5195	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
5165	1,1,2-Trichloroethane
4630	1,1-Dichloroethane
4640	1,1-Dichloroethylene
5155	1,2,4-Trichlorobenzene
5210	1,2,4-Trimethylbenzene
4570	1,2-Dibromo-3-chloropropane (DBCP)
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
4695	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane (Ethylene dichloride)
4655	1,2-Dichloropropane
5215	1,3,5-Trimethylbenzene
9318	1,3-Butadiene

ORELAP Fields of Accreditation

ORELAP ID: 4068
EPA CODE: CA01627
Certificate: 4068 - 001

ALS Environmental - Simi Valley

2655 Park Center Drive, Suite A
Simi Valley CA 93065

Issue Date: 02/16/2015 ***Expiration Date:*** 02/15/2016

As of 02/16/2015 ***this list supercedes all previous lists for this certificate number. Customers. Please verify the current accreditation standing with ORELAP.***

Analyte Code	*Analyte*
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
4735	1,4-Dioxane (1,4- Diethyleneoxide)
5220	2,2,4-Trimethylpentane
4410	2-Butanone (Methyl ethyl ketone, MEK)
4860	2-Hexanone (MBK)
4995	4-Methyl-2-pentanone (MIBK)
4315	Acetone
4320	Acetonitrile
4375	Benzene
4395	Bromodichloromethane
4400	Bromoform
4450	Carbon disulfide
4455	Carbon tetrachloride
4475	Chlorobenzene
4575	Chlorodibromomethane
4485	Chloroethane (Ethyl chloride)
4505	Chloroform
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4555	Cyclohexane
4625	Dichlorodifluoromethane (Freon-12)
4750	Ethanol
4765	Ethylbenzene
4835	Hexachlorobutadiene
4895	Isopropyl alcohol (2-Propanol, Isopropanol)
4900	Isopropylbenzene
5240	m+p-xylene
4960	Methyl chloride (Chloromethane)
5000	Methyl tert-butyl ether (MTBE)
4975	Methylene chloride (Dichloromethane)
5005	Naphthalene
4825	n-Heptane
4855	n-Hexane
5027	n-Octane
5250	o-Xylene
5100	Styrene
5115	Tetrachloroethylene (Perchloroethylene)
5120	Tetrahydrofuran (THF)
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
5235	Vinyl chloride
5260	Xylene (total)

EPA TO-4A **10249204** **Pesticides and PCBs by HV PUF GC**

Analyte Code	*Analyte*
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
8880	Aroclor-1016 (PCB-1016)
8910	Aroclor-1260 (PCB-1260)
7115	beta-BHC (beta-Hexachlorocyclohexane)
7105	delta-BHC
7470	Dieldrin

ORELAP Fields of Accreditation

ORELAP ID: 4068
EPA CODE: CA01627
Certificate: 4068 - 001

ALS Environmental - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley CA 93065

Issue Date: 02/16/2015 ***Expiration Date:*** 02/15/2016

As of 02/16/2015 ***this list supercedes all previous lists for this certificate number. Customers. Please verify the current accreditation standing with ORELAP.***

Analyte Code	Analyte
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide
7810	Methoxychlor

MADEP APH **90017188** **Air-Phase Petroleum Hydrocarbons**

Analyte Code	Analyte
9318	1,3-Butadiene
3792	APH Aliphatics C5-C8
3793	APH Aliphatics C9-C12
3794	APH Aromatics C9-C10
4375	Benzene
4765	Ethylbenzene
5240	m+p-xylene
5000	Methyl tert-butyl ether (MTBE)
5005	Naphthalene
5250	o-Xylene
5140	Toluene

ALS Standard Operating Procedure

Proprietary - Uncontrolled Copy

DOCUMENT TITLE:	ANALYSIS OF HALOGENATED VOLATILE ORGANIC COMPOUNDS IN EMISSIONS FROM STATIONARY SOURCES USING GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTION (ECD) IN ACCORDANCE WITH A MODIFICATION OF CARB METHOD 422
REFERENCED METHOD:	CARB 422 MODIFIED
SOP ID:	SVO-CARB422
REV. NUMBER:	05.0
EFFECTIVE DATE:	04/25/2015

STANDARD OPERATING PROCEDURE

ANALYSIS OF HALOGENATED VOLATILE ORGANIC COMPOUNDS IN EMISSIONS FROM STATIONARY SOURCES USING GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTION (ECD) IN ACCORDANCE WITH A MODIFICATION OF CARB METHOD 422

CARB 422 MODIFIED

SOP ID:	SVO-CARB422	Rev. Number:	05.0	Effective Date:	04/25/2015

Approved By: _____ Date: 4/14/15
SVOC Team Leader – Madeleine Dangazyan

Approved By: _____ Date: 4/20/15
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Approved By: _____ Date: 4/15/15
Laboratory Director – Kelly Horiuchi

Archival Date:	_____	Doc Control ID#:	Non-Controlled	Editor:	_____

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ANALYSIS OF HALOGENATED VOLATILE ORGANIC COMPOUNDS IN EMISSIONS FROM STATIONARY SOURCES USING GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTION (ECD) IN ACCORDANCE WITH A MODIFICATION OF CARB METHOD 422

1) Scope and Applicability

1.1 This gas chromatograph method is used in the analysis of chloroform, trichloroethene, and tetrachloroethene by a modification of CARB Method 422. Other compounds that maybe reported provided that the requirements of this document are followed are: carbon tetrachloride, 1,2-dichloroethane, 1,2-dibromoethane, trichlorofluoromethane, 1,3-butadiene and dichloromethane. This method cannot be used to determine compounds of high molecular weight, compounds that may polymerize before analysis or compounds that have very low vapor pressures at stack or instrument conditions.

1.2 This method applies to but is not limited to the following sample matrices: ambient air, source emissions, landfill gases, digester gases, and vehicular exhaust. The range of this method for quantifying target analyte gases, depending on the concentration of the samples, is approximately 0.0010 to 200ppm. The upper limit may be extended by diluting the sample with an inert gas or by using a smaller injection volume. Approximately twenty samples may be analyzed in one eight hour day.

2) Summary of Procedure

2.1 Samples are collected in Tedlar bags, and delivered to the laboratory for analysis. A modification of the method may be used for the collection of samples in Summa canisters or glass bottles. An aliquot is drawn from the sampling container using a gastight syringe and injected onto a chromatographic column where the analytes are separated and measured using an electron capture detector (ECD). Analytes are identified and quantified based on their retention time, which is compared with that of a known standard under identical conditions. The Tedlar bag sampling and analysis is not suitable for monitoring 1,3-butadiene in combustion source emissions. Refer to CARB Method 422.102 for the analysis of 1,3-butadiene.

3) Definitions

3.1 Relative Standard Deviation (RSD) The RSD is the coefficient of variation (CV; ratio of the standard deviation to the mean) multiplied by 100 to convert the CV to a percentage of the mean.

3.2 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.3 Field Sample A sample collected and delivered to the laboratory for analysis.

3.4 Batch QC Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD), etc.

3.5 Calibration Standard (Initial Calibration – ICAL) A calibration standard is a known concentration of desired analyte(s) prepared from a primary standard, which is, in turn, prepared from a stock standard material. A calibration standard is analyzed at varying concentrations and used to calibrate the response of the measurement system with respect to analyte concentration.

3.6 Initial Calibration Verification (ICV) Standard An initial calibration verification standard (ICV) is a standard that is prepared from materials obtained from a source other than

the source for the calibration standards and is analyzed after the measurement system is calibrated, but prior to sample analysis in order to verify the calibration of the measurement system.

3.7 Continuing Calibration Verification (CCV) Standard A continuing calibration verification standard (CCV) is a midrange calibration standard that is analyzed periodically to verify the continuing calibration of the measurement system.

3.8 Method Blank (MB) The method blank (MB) for this method is ultra-pure nitrogen that is analyzed to verify the zero point of the analytical system and to verify freedom from carryover.

3.9 Method Reporting Limit (MRL) The minimum reliably quantifiable concentration of a compound.

3.10 Laboratory Control Sample (LCS) For the purposes of this document, a laboratory control sample (LCS) shall be a calibration standard of known concentration. The percent recovery of the analyte(s) in the LCS is used to assess method performance.

3.11 Laboratory Duplicate Aliquots of a sample taken from the same container under laboratory conditions which are processed and analyzed independently.

3.12 Precision Precision of a method is how close results are to one another, and is usually expressed by measures such as standard deviation, which describe the spread of results.

3.13 Bias The bias of a method is an expression of how close the mean of a set of results (produced by the method) is to the true value.

3.14 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop “ticks” have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

4) Health and Safety Warnings

4.1 Pollution Prevention and Waste Management All waste management must be carried out in accordance with the most recent version of the *SOP for Waste Disposal*.

4.2 This procedure may include CHEMICAL, OPERATIONAL and/or EQUIPMENT hazards. Employees must review and understand the following hazards and their preventive measures prior to proceeding with this activity. Hazard information related to this activity which is not included or referenced in this document should be immediately brought to the attention of the Department Supervisor.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Standard and sample preparation.	Exposure to potential health hazards through absorption through skin. Inhalation hazards.	Reduce exposure through the use of gloves and fume hoods. Safety glasses must be worn when working in the prep lab. Care should be taken when handling standard material in a neat or highly concentrated form. Personal protective clothing (safety glasses, gloves, and lab coat) are required when handling standard material in neat form. Consult Safety Data Sheets (SDS) for compounds being handled in this procedure, and be familiar with proper safety precautions. SDS shall be reviewed as part of employee training. Refer to the laboratory's *Environmental Health and Safety Manual* for additional information regarding safety in the workplace.
Job Task #2:	**Hazards**	**Preventative Measures**
Using and moving compressed gas cylinders.	Gas leak, fire, and explosion. Personal injury due to falling during transport.	All cylinders must be secured in an upright position to a wall or immovable counter with a chain or a cylinder clamp when not in use. Keep safety caps on when cylinders are not in use. A handcart must be used when transporting cylinders. The cylinder must be secured to the handcart with a chain or belt. Full cylinders must be kept separate from empty cylinders. Flammable gases (i.e. pressurized hydrogen) must be clearly labeled. Flammables and oxidizers must be separated by a ½-hour fire wall or by at least twenty feet.
Job Task #3:	**Hazards**	**Preventative Measures**
Glass syringe use	Skin lacerations and punctures.	The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.
Job Task #4:	**Hazards**	**Preventative Measures**
Working with and Pressurization of glass bottles	Personal injury from breakage or shattering.	Wear safety glasses when working with glass bottles. Gloves may be worn to help maintain grip. Bottle Vacs must not be pressurized higher than 7 psig.

5) Cautions

5.1 An instrument maintenance log shall be kept documenting maintenance performed on each analytical system. This log must be kept current. The serial numbers of each instrument shall be recorded in the front of the logbook. An entry shall be made in the appropriate log every time maintenance is performed. The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, or cleaning the source. The entry in the log must include:

(a) the date of maintenance
(b) who did the maintenance
(c) description of the maintenance
(d) proof that the maintenance activity was successful

A notation of a successful continuing calibration or initial calibration shall serve as proof that the maintenance is complete and the instrument is in working order.

5.2 Carrier Gas Purifier

If in-line purifiers or scrubbers are in place, these purifiers must be changed as recommended by the supplier.

5.3 GC System

5.3.1 Column Performance should be monitored by observing peak shapes and column bleed. Over time, the column may exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur depends on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Poor performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.3.2 Injection Port Injection port maintenance includes changing the injection port liner and column ferrule as needed. Liners should be changed when recent sample analyses predict a problem in chromatographic performance.

5.3.3 Injector Septa Septa should be changed monthly or whenever there is a noticeable change in peak definition. For best results with air analyses, two septa are placed into the injector in order to eliminate loss during manual injections.

5.3.4 Electron Capture Detector The ECD contains Nickel-63 and must undergo a radioactive leak or “wipe” test every 6 months. The radioactive leak test records are to be maintained for a minimum of 3 years. If a leak test fails, the ECD must be immediately taken out of use and the following must occur:

- Send detector to an authorized repair or disposal facility.
- Prepare and submit a report within 30 days to the California department of radiological health including a complete description of the device (manufacturer, type, serial number) and a brief description of the event and the remedial action taken (*California Code of Regulations).*

Under no circumstances is the ECD unit to be opened, cleaned, repaired or modified by laboratory personnel, as this would be a direct violation of the General License requirement.

6) Interferences

6.1 Contaminated Sample

Care must be taken to prevent ambient air intrusion into the sample container during canister pressurization and laboratory analysis. When using adapters and fittings the dead volume must be evacuated and replaced with the sample gas prior to sampling from the container. The sampling syringe shall then be flushed with the sample gas to remove residual ambient air. An aliquot greater than is needed is drawn, and the syringe plunger is adjusted to the appropriate volume *immediately* before injecting.

6.2 Carrier Gas Contamination

To prevent system contamination, UHP/ZERO grade helium (99.999% purity) is used as the carrier gas. Additionally, a purifier is incorporated into the analytical system as another precaution in preventing contamination.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review and reporting. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP must perform analysis and interpretation of the results. This demonstration shall be in accordance with the training program of the laboratory. The department supervisor/manager or designee shall perform final review and sign-off on the data.

7.2 Training shall be conducted in accordance with the *SOP for Training Policy.* An initial demonstration of proficiency shall be performed prior to independent analyses of samples. In addition, a continuing demonstration must be performed annually or prior to the analysis of samples. Both demonstrations consist of spiking Tedlar bags with the LCS standard and evaluating for both precision and accuracy. The criteria for approval are the same as the acceptance criteria for the LCS as specified in this document. See Attachment 1.

8) Sample Collection, Handling, and Preservation

8.1 The samples are collected and delivered to the laboratory for analysis in either Tedlar bags or specially prepared canisters, or glass sampling bottles (Bottle Vac. Entech Instruments). Samples collected in bags must be analyzed within 72 hours after sample collection unless otherwise specified by the client. Samples delivered in cleaned, evacuated summa or other specially prepared canisters do not have specified holding times for atmospheric gases but should be analyzed within 30 days from the date of collection.

9) Equipment and Supplies

9.1 Gas Chromatograph

HP 5890 or equivalent equipped with an electron capture detector, and having a temperature programmable oven. The column shall be 60m, 0.53mm ID RT_x-1 or equivalent with a 5µm film thickness.

Conditioning of the chromatographic column is required prior to use of the system. The column should be conditioned with a continuous flow of chromatographic grade helium and temperature programmed from 35°C to 200°C at a rate of five degrees per minute. The column should be held at 200°C for at least four hours.

9.2 Regulators

Regulators are used on the gas cylinders supplying the GC and for preparing cylinder standards.

9.3 Data System

A data system with the ability to collect data from the GC detector, integrate the peaks and perform the appropriate quantification calculations shall be used. This laboratory currently uses HP Chemstation/Enviroquant GC software.

9.4 Syringes

Gas tight syringes of the following volumes: 10mL, 1.0mL, and 0.5mL.

9.5 Tedlar Bags

New Tedlar bags are used for preparing standards and diluting very concentrated samples, which fall outside of the initial calibration range.

10) Standards and Reagents

10.1 All samples and standards must be stored separately. The concentration, preparation and expiration date as well as analyst's initials must be identified on the standard label. Each standard must also be uniquely identified with a laboratory ID number.

All certificates shall be noted with the standard identification number, date received and initials of the receiving analyst and retained by the quality assurance department.

10.2 Carrier and Calibration Standard Balance Gas

10.2.1 Helium - UHP/ZERO (99.999%) or higher in purity

10.2.2 Nitrogen - UHP/ZERO (99.999%) or higher in purity

10.3 Neat Standards

These standards must be stored in accordance with the requirements described in the *SOP for Handling Consumable Materials*. These standards may be stored for a period of 5 years for neat standards, 2 years for air standards or as recommended by the manufacturer.

Compound	Purity	MW	Density
Chloroform	99+%	119.4	1.4832
Trichloroethene	99+%	131.4	1.4642
Tetrachloroethene	99+%	165.8	1.6227
Carbon tetrachloride	99+%	153.8	1.5940
1,2-Dichloroethane	99+%	98.96	1.2351
1,2-Dibromoethane	99+%	187.9	2.17
Trichlorofluoromethane	99+%	137.4	1.494
1,3-Butadiene	99+%	54.09	0.6149
Dichloromethane (Methylene Chloride)	99+%	84.94	1.3266

10.4 Initial Calibration Standard / Working Standard

Prepare a neat cocktail standard from the above stated compounds by adding the appropriate volume of the neat compounds into a clean 2mL vial.

The current cocktail concentration recommendation is 200ppm for tetrachloroethene and 1000ppm for all other compounds. Determine the spike volume and add this amount to 1L of high purity nitrogen in a Tedlar bag. Record the calibration standard in accordance with the requirements described in the *SOP for Handling Consumable Materials.* Depending on the desired dynamic range of the initial calibration, various dilutions shall be made from this standard bag. A serial dilution may also be prepared from this standard bag.

The intermediate standard, along with all dilutions, must be stored at room temperature and expires 3 days after preparation.

10.4.1 *Equi-mass "soup"* (contains compounds in equal mass amounts) or *cocktail* prepared from neat compounds.

Cocktail Preparation:

Step 1: This cocktail is prepared by combining a calculated amount of each neat compound into a small glass vial based on the desired Tedlar bag standard concentration of 200ppm for tetrachloroethene and 1000ppm for other compounds. Use a microliter syringe to transfer each compound, cleaning with solvents in between. Put the vial in the freezer between aliquots to minimize volatilization. Take the density and molecular weight of each compound into account to determine the actual amount of each compound to spike into the cocktail by using the following equation.

$$S = \frac{\frac{S_A * MW * 1L * \frac{m^3}{1000L}}{24.46}}{D} \qquad \text{(Equation 1)}$$

Where:

S Calculated volume, per compound (uL)
MW Molecular weight for each compound, g/mole
S_A Desired concentration for each compound (ppm= mg/m³)
D Density (g/mL); refer to the density references

Example: The actual volume of chloroform to add to the cocktail is calculated by the following.

$$\text{S(Chloroform)} = \frac{\dfrac{1000mg/m^3 * 119.4 * 1L * \dfrac{m^3}{1000L}}{24.46}}{1.4832} = 3.29\text{uL}$$

Hint: To obtain a larger cocktail volume, multiply each compound volume by a multiplier (i.e., 40 or 50). This procedure prevents from having to prepare cocktail more often.

Tabulate all of the calculated spike amounts and spike the total into a 1L Tedlar bag filled with nitrogen. Place in an oven at approximately 60ºC for about 10 minutes. Allow the Tedlar bag to sit for about 15-20 minutes for an equilibrium period.

11) Method Calibration

11.1 Initial Calibration

The instrument must be calibrated initially and whenever the laboratory takes corrective action (maintenance), which may change or affect the initial calibration criteria, or if the continuing calibration acceptance criteria have not been met. Introduce each initial calibration concentration standard (at least five levels, analyzed from low concentration to high concentration) by direct injection using a gas tight syringe. Perform all calibration runs according to the analytical portion of the sample analysis described in Section 12.1

Note: The concentrations of the initial calibration may change as long as the low standard analyzed is the same as the reporting limit for each analyte.

11.1.1 Initial Calibration Requirements

Once a set of ICAL standards is analyzed, the previous ICAL may no longer be used to analyze new samples and it must be archived. The only time an archived ICAL can be used thereafter is to review or re-evaluate samples(s) previously processed using that ICAL.

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. Highest concentration, together with the lowest concentration, defines the calibration curve.
3. Lowest concentration must be at method reporting limit.
4. A blank should be analyzed prior to beginning the analysis of the calibration standards.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from low to high concentration.
8. All ICAL analyses must be completed within 48 hours.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.

- Lowest concentration must be at the MRL and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
- Points at high end may be dropped, but doing so lowers the calibration curve range.
- Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution or standard preparation error, or instrument malfunction) is accounted for and documented in a nonconformity and corrective action report (NCAR). In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action.
- If a point or a calibration standard is dropped, the reason must be documented (and the results maintained with the documentation for the final ICAL).
- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 48 hours).
- Once the ICAL has been used to calculate and report sample results, it is not to be changed.

11.1.2 Initial Calibration Review

Analyst's calculations and assessment along with a peer review of all ICAL data and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples. Sample results may only be reported if the ICAL is reviewed and found to be acceptable.

11.1.3 Initial Calibration File

An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- Blank analysis quantitation report
- Calibration status report (a.k.a. Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and evaluate continuing calibration report (a.k.a. Percent Difference Report)

11.1.4 Initial Calibration Verification

Verify the initial calibration by analyzing an independent calibration verification standard (ICV).

12) Sample Preparation/Analysis

12.1 Analytical Sequence and Data System Setup

12.1.1 Data System Load the appropriate acquisition method file for the gas chromatograph temperature program. Load the appropriate analytical sequence. Enter the analytical sequence information in the table window, including standard name, sample name and injection volume. Run the sequence and inject the standards and samples per the guidelines in Section 12.1.2.

12.1.2 Analytical Sequence The analytical batch must be completed for the analysis of ≤20 field samples. Laboratory duplicates (LD), duplicate field samples and sample dilutions are considered samples. Batch QC samples may be analyzed anywhere in the analytical sequence, with the exception of the method blank which must be analyzed prior to sample analysis in order to demonstrate a contamination free system.

Analytical Sequence Guideline[1]

Sample Description(w/ICAL)	*Sample Description*
Calibration Stds.[2]	CCV[3]
ICV[4]	MB[5]
MB[5]	LCS[6]
LCS[6]	Samples 1-10
Samples 1-10	CCV[3]
CCV[3]	Samples 11-19
Samples 11-19	LD[7]
LD[7]	CCV[3]
CCV[3]	

[1] The batch QC may be analyzed in an order other than the one listed in this document; the analytical sequence specified below is a guideline.
[2] The initial calibration must be generated in accordance with the guidelines detailed in Section 11.1 of this document.
[3] In cases, where the ICAL is not performed the analytical sequence must begin with the analysis of a CCV standard. In addition, the analytical sequence shall end with an acceptable CCV.
[4] Every ICAL must be followed by a second source standard (ICV) which contains all of the target analytes.
[5] The method blank must be analyzed prior to any samples within the sequence.
[6] Every analytical sequence must include a laboratory control sample. A LCS shall be analyzed at a rate of one per twenty samples or fewer for each analyte.
[7] A laboratory duplicate must be analyzed at a frequency of 1 in 20 or fewer samples.

12.2 GC Configuration

12.2.1 Temperature Program The GC oven temperature programming must be set to completely elute all of the target analytes. The temperature program ramps up to a high temperature, not exceeding the maximum temperature rating of the column in use, and holds there to allow all heavier compounds to elute, in order to prevent carryover to the next injection. The settings and system parameters are as follows.

Instrument Control Parameters – GC21	
Sample Inlet	GC
Injection Source	Manual
Run Time	12 minutes
Injector	
Mode	Packed
Temperature	150˚C
Pressure	18psi at 100˚C oven temperature
Isothermal Oven	
Initial Temperature	100˚C
Initial Time	12 minutes
Column	
Model Number	RTx-1
Nominal Length	60m
Nominal Diameter	0.53mm ID
Film Thickness	5µm
ECD	
N2	50mL/min
Temperature	280˚C

12.3 Continuing Calibration

A continuing calibration check shall be performed at the beginning, after every 10 samples and at the end of an analytical sequence, or every twenty field samples, not to exceed a 24-hour period. The concentration of the calibration verification may be varied within the established calibration range.

12.4 Method Blank

The method blank shall be obtained using ultra high purity nitrogen directly injected in the same manner as the standards and samples. A method blank must be analyzed prior to analysis of samples. A method blank must also be analyzed if carryover contamination is suspected.

12.5 Laboratory Control Sample

The laboratory control sample shall be an injection of the continuing calibration or initial calibration verification standard. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

12.6 Analysis

12.6.1 Canister and Glass Bottle Pressurization Sample analysis must be made using the same instrument parameters as that of the calibration standards. Refer to the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters* for the procedure of how canisters and glass bottles are to be pressurized prior to analysis. The analyst shall record the appropriate pressures on the Service Request form. This includes noting any anomalies for which the appropriate corrective actions have been detailed and must be followed accordingly.

12.6.2 Sample Analysis Sample analysis shall be performed by a direct injection technique using gas tight syringes. Insert the syringe through the Tedlar bag septum or summa can fit with an adapter. When using adapters and fittings the dead volume must be evacuated and replaced with the sample gas prior to

sampling from the container. The sampling syringe shall then be flushed with the sample gas to remove residual ambient air and vented into a waste bag. This procedure entails drawing an aliquot greater than is needed, and adjusting the syringe plunger to the appropriate volume *immediately* before injecting.

Note: The maximum allowed injection volume is 500uL

Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments). Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fittings higher than 80ºC.

12.6.3 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot or a dilution in a Tedlar bag must be analyzed. Guidance in performing dilutions and exceptions to this requirement are given below.

- Use results of the original analysis to determine the approximate dilution factor required getting the largest analyte peak within the initial calibration range.
- The dilution factor chosen should keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument. Additional compounds may be reported as long as they are within the calibration range.
- Analysis involving dilution should be made with high purity nitrogen and must be reported with a dilution factor.

Tedlar bag dilution:

- Calculate the sample amount and volume of balance gas needed to obtain the required dilution.
- Fill a new 1.0L Tedlar bag with nitrogen using the appropriate gas tight syringe.
- Remove the difference in the balance gas using the appropriate gas tight syringe.
- Add the calculated sample amount using a gas tight syringe.

12.7 Laboratory Duplicate

Analyze two separate aliquots from the same sample container. A laboratory duplicate must be analyzed a frequency of 1 in 20 field samples. The laboratory duplicate should be rotated among clients, whenever possible

12.8 Manual Integration

The integration(s) for each sample is checked to ensure that it has been integrated properly. Assuming an incorrect automatic integration the analyst shall conduct the manual integration in accordance with the *SOP for Manual Integration Policy* including all documentation and reviews associated with the process. The review should include the analyst and peer reviewer initialing and dating the manual integration as an indication of acceptability and approval.

12.9 Method Detection and Quantitation Limits

The MDL must be performed in accordance with the procedure outlined in the *SOP for the Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* Method detection limits must be determined annually and whenever

there is a change in the test method that affects how the test is performed, or when a change in instrumentation is such that it affects the sensitivity of the analysis. The MDL study shall be performed on each instrument for which this method is performed. All supporting data must be approved and retained.

12.10 Cleaning Tedlar Bags

Fill with nitrogen and evacuate several times. In the final cleaning step partially fill the bags with nitrogen and evacuate using a pump.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. Files should be named with a character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files shall be saved in a unique sub-directory on the server. An example of how the analyst should store analytical data is as follows:

Instrument Number/Data/Method ID/yr_month/.d*

The initial calibration curve may be saved with an identification such as CARB followed by the date of the analysis (mm,yy). This file should be saved in the following directory: J:\"instrument ID"\Method\. No curve may be overwritten at any time to ensure a complete audit trail.

14.2 Sufficient raw data records must be retained of the analysis, instrument calibrations and method detection limit studies including: analysis/calibration date and time, test method, instrument, sample identification, each analyte name, analyst's initials, concentration and response, and standards used for the analysis and calibrations, any manual calculations including sample dilutions and manual integrations. Information entered and reported on the quantitation report and instrument run log must be complete and accurate.

14.3 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date of analysis, time of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, any manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings), standard and reagent origin, sample receipt, calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, and assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 Initial Calibration

- Tabulate the linear relationship using Equation 1

15.2 Initial Calibration Verification

- Calculate the concentration for each analyte using equation number 1.
- Calculate the percent difference (%D) between the calculated concentration (equation number 1) and the actual concentration using equation number 2.

15.3 Continuing Calibration Verification

- Calculate the concentration of each analyte using equation number 1.
- Calculate the percent difference (%D) between the calculated concentration (equation number 1) and the actual concentration using equation number 2.

15.4 Laboratory Control Sample

- Calculate the concentration of each analyte using equation number 1.
- Calculate the percent recovery (%R) for each analyte using equation number 4.

15.5 Sample Analysis

- Calculate the concentration of each analyte using equation number 1.
- Calculate the dilution factor if necessary using equation number 5.

15.6 Laboratory Duplicate

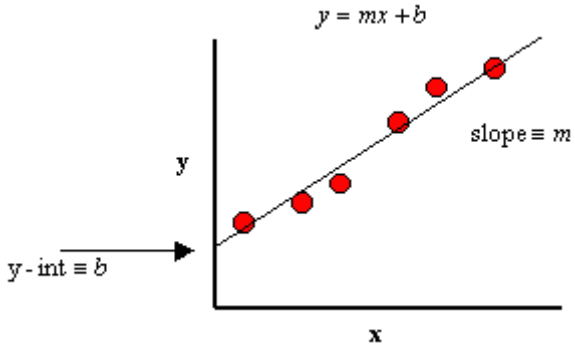
- Calculate the concentration of each analyte using equation number 1.
- Calculate the relative percent difference (RPD) using equation number 3.

15.7 Calculations

15.7.1 Equation Number 1

Linear Relationship

Say we have a set of data, (x_i, y_i), shown at the left. If we have reason to believe that there exists a **linear relationship** between the variables **x** and **y**, we can plot the data and draw a "best-fit" *straight line* through the data. Of course, this relationship is governed by the familiar equation $y = mx + b$. We can then find the **slope, *m*,** and **y-intercept, *b*,** for the data, which are shown in the figure below.



Linear Regression Equations

If we expect a set of data to have a linear correlation, **it is not necessary for us to plot the data** in order to determine the constants *m* (slope) and *b* (y-

intercept) of the equation $y = mx + b$. Instead, we can apply a statistical treatment known as **linear regression** to the data and determine these constants.

Given a set of data (x_i, y_i) with *n* data points, the slope, y-intercept and correlation coefficient, *r*, can be determined using the following: (Note that the limits of the summation, which are *i* to *n*, and the summation indices on *x* and *y* have been omitted.)

$$m = \frac{n\sum(xy) - \sum x \sum y}{n\sum(x^2) - (\sum x)^2}$$

$$b = \frac{\sum y - m\sum x}{n}$$

$$r = \frac{n\sum(xy) - \sum x \sum y}{\sqrt{\left[n\sum(x^2) - (\sum x)^2\right]\left[n\sum(y^2) - (\sum y)^2\right]}}$$

Casio fx-300W Calculation Instruction for Linear Regression:

The regression formula for linear regression is: y = A+Bx
Enter REG Mode (Linear Regression)
Hit Mode, 3 Reg, 1 Lin shift Scl = (memory clear)
Enter data points - concentration vs absorbance: [ex: 0,0 'M+', 0.1, 0.099 'M+', 0.2, 0.198 'M+' & 0.4, 0.402 'M+']
For correlation coefficient – hit 'Shift' then 'r' then '=' 0.999965116
For unknown concentrations enter absorbance (ex: 0.175 from spectrophotometer) - hit shift '+' key = 0.17524865

15.7.2 Equation Number 2

Percent Difference, %D,

The %D is used for evaluating ICV and CCV vs. the initial calibration

$$\%D = \frac{C_{CCVorICV} - C_{std}}{C_{std}}(100)$$

where, for any given analyte:

$C_{CCVorICV}$	is the concentration being evaluated
C_{std}	is the concentration from the current calibration curve

15.7.3 Equation Number 3

Relative Percent Difference (RPD)

$$\frac{|R_1 - R_2|}{\left(\frac{R_1 + R_2}{2}\right)} x100$$

where:

R_1 First measurement value
R_2 Second measurement value

15.7.4 Equation Number 4

Percent Recovery (%R):

$$\%R = \frac{C}{S} x100$$

Where:

C = Concentration of the analyte recovered
S = Spiked amount

15.7.5 Equation Number 5

Dilution Factor

$$DF = \frac{V_T}{V_S}$$

Where:

DF = dilution factor
V_S = volume of sample (mL) used
V_T = total volume of dilution (mL)

15.7.6 Equation Number 6

Results

In order to obtain the final reported value, the result must be adjusted with the canister dilution factor, any sample dilution and injection volume and converted to ug/m^3.

Example:

- R = Result = 22.079ppb (on column)
- DF = Dilution Factor = 1.58 (canister dilution factor)
- IV_N = Normal Injection Volume = 0.5mL (see method blank injection volume)
- IV_A = Actual Injection Volume = 0.1mL
- AD = Additional Dilution = 1000

$$ppbV = \frac{R * DF * IV_N * AD}{IV_A} = \frac{22.079 * 1.58 * 0.5 * 1000}{0.1} = \frac{17442}{0.1} = 174424 =$$

170,000ppbV

- MW is the molecular weight of PCE
- 24.46 is the molar volume of gas at lab conditions (constant)
- All results are reported with two significant figures

$$ug/m^3 = \frac{ppbV * MW}{24.46} = \frac{174424 * 165.8}{24.46} = 1{,}182{,}318 = 1{,}200{,}000 ug/m^3$$

15.8 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated following the data review checklist in Attachment 3. The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The data review checklist shall be used to document the review process. Once it has been completed, the checklist must be initialed, dated and filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.3.

15.9 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results. The analyst shall ensure that all of the requirements specified in this document and the *SOP for Data Review and Reporting* are followed.

15.10 Sample Preparation and Analysis Observations / Case Narrative Summary Form

The case narrative summary form, which is included in the *SOP for Laboratory Storage, Analysis, and Tracking*, must be generated when there are any specific sample composition information, sample preparation, analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved.

This form is necessary for documentation purposes and shall be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through preparation, analysis, data reduction, and reporting.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 This section contains technical acceptance criteria. To the extent possible, samples shall be reported only if all of the quality control measures are acceptable.

16.2 It must be determined if there are any instrumentation problems contributing to the occurrence of any out-of-control data. If it is decided that problems do exist, then the analyst must determine if the effects have caused any modification in the data from

client submitted samples. This being the case, all samples (including QC) that are affected by instrumentation problems must be re-analyzed following any necessary maintenance activity. All corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate.

16.3 Initial Calibration

16.3.1 Acceptance Criteria

- The correlation coefficient must be at least 0.98 from the least squares fit for the calibration to be considered acceptable.
- The retention time of each analyte at each calibration level must be within 0.1 minute of the midpoint standard in the calibration curve.

16.3.2 Corrective Action Inspect the system for possible sources. It may be necessary to change the column or take other corrective actions. Also, check standards for a bad injection and re-analyze standard. If a bad injection is not evident, perform maintenance and attempt another initial calibration (make notation in maintenance logbook regarding any steps taken). A demonstration of an in-control system is required before proceeding with the analysis.

Note: No ICAL may be interrupted by any maintenance procedure. Therefore, all standards incorporated in a curve must be reanalyzed.

16.4 Initial Calibration Verification Standard (ICV)

16.4.1 Acceptance Criteria

- The percent difference (%D) for each calculated target analyte must be within ±30% of the actual concentration of the standard.
- The retention time of each target analyte must be within 0.1 minute of the midpoint standard in the calibration curve.

16.4.2 Corrective Action The initial calibration verification should be re-analyzed. A second failed ICV must initiate corrective action and two consecutive ICVs must pass in order for the ICAL to be deemed acceptable. It may be necessary to prepare either new ICAL or ICV standards or both, perform maintenance and reanalyze the initial calibration.

16.5 Continuing Calibration Verification (CCV)

16.5.1 Acceptance Criteria

- The percent difference (%D) for each calculated target analyte must be within ±30% of the actual concentration.

16.5.2 Corrective Action If the criteria are not met, reanalyze (no more than two injections may be made before corrective action is initiated) or prepare a fresh CCV standard and reanalyze. If routine corrective action procedures fail to produce an acceptable calibration verification, a new initial calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data only under the following special condition:

When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the sample affected by the unacceptable CCV shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

16.6 Method Blank

16.6.1 Acceptance Criteria

- The method blank result for any target analyte must not be greater than the method reporting limit or contribute more than 10% of the sample concentration.

16.6.2 Corrective Action The source of the problem must be investigated and measures taken to eliminate the cause. Determine whether the contamination is from the instrument or due to contamination in the nitrogen, syringe or other source. Regardless, appropriate corrective measures must be taken and documented before further sample analysis proceeds. If the results are the same, the blank along with all associated samples must be reported to the client with the appropriate qualifier.

16.7 Laboratory Control Sample (LCS)

16.7.1 Acceptance Criteria

- The percent recovery for all compounds must be within 70% and 130%.

16.7.2 Corrective Action Determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and reanalyze the associated sample(s). If the problem is with the injection, reanalyze the LCS. If the results are still unacceptable and there does not appear to be any instrumentation problems refer to the appropriate reporting information.

16.8 Sample Analysis

16.8.1 Acceptance Criteria

- Sample results must be quantitated from the current instrument initial calibration and may not be quantitated from any continuing calibration verification standard.
- The field samples must be analyzed along with a laboratory method blank that has met the method blank criteria.
- All target analyte peaks must be within the initial calibration range.
- The retention time of each target analyte must be within 0.1 minute of the CCV.

16.8.2 Corrective Action To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out-of-control, and the data must be reported, all samples associated with the out-of-control quality control measures shall be reported with the appropriate data qualifier(s).

- When corrective actions are made, samples analyzed while the system was not functioning properly must be reanalyzed.
- Results not bracketed by initial instrument calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags.

16.9 Laboratory Duplicate

16.9.1 Acceptance Criteria

- The selected samples must be rotated among client samples so that various matrix problems may be noted and/or addressed.

- The results must meet all of the criteria for sample analysis as well as be <15% relative percent difference for all analytes of interest, provided that the concentration is greater than 10x the RL.

16.9.2 Corrective Action The sample(s) should be re-analyzed whenever the duplicate results are outside the technical acceptance window. If the results are still unacceptable and there does not appear to be any matrix effects, interfering peaks, or instrument problems, the results for both injections shall be reported to the client with the appropriate qualifier.

16.10 Samples Holding Time Expired The client is to be notified (best attempt) that the sample's holding time was missed and the client is to decide if the sample analysis shall continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 If a quality control measure if found to be out-of-control and the data must be reported, all samples associated with the out-of-control quality control measure shall be reported with the appropriate data qualifier(s).

18.2 Analysis quality control results (CCV, MB, LD, and LCS recoveries) out-of-control

If the associated samples are within holding time, re-analyze the sample. Alternatively, evaluate the effect on the sample results and report the results with qualifiers and/or discuss in the case narrative as detailed below.

18.2.1 CCV The LCS should be in control in order for any results to be reported with an out-of-control CCV (biased high). Refer to Section 16.5.

18.2.2 Method Blank If an analyte in the blank is found to be out-of-control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report. If the analyte is found in the blank but not in the sample and all other quality control meets acceptance criteria then the results for the sample may be reported without a qualifier. However, if other QC is out-of-control then an evaluation must be made and the results reported accordingly.

18.2.3 Laboratory Control Sample If the samples are analyzed with an out-of-control LCS, then all reported analytical results must be "flagged" with the appropriate data qualifier and/or discussed in the case narrative.

18.2.4 Laboratory Duplicate The appropriate data qualifier must be included for results associated with an out-of-control laboratory duplicate and/or discussed in the case narrative.

18.3 Sample quality control results out-of-control

Examine the sample results for matrix interference and for carryover. Reanalyze the sample(s) and/or reanalyze the sample(s) at a lower aliquot. If the out-of-control

results are due to matrix interference, report the results with a matrix interference qualifier.

Holding time qualifiers must be reported on samples not analyzed within holding time.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
05.0	04/25/15	C. Humphrey	Section 1.1 – Removed 1,1,1-trichloroethane
			Section 2.1 – Added glass bottles
			Section 4 – Revised
			Section 8.1 – Added glass bottles
			Section 10.3 – Removed 1,1,1-trichloroethane
			Section 12.6.1 – Revised; added glass bottles
			Table 1 – Removed 1,1,1-trichloroethane; updated MDL values

21) References and Related Documents

21.1 State of California Air Resources Board, Method 422 "Determination of Volatile Organic Compounds in Emissions from Stationary Sources", Amended December 13, 1991.

21.2 *SOP for Making Entries onto Analytical Records*, SOP ID CE-QA007

21.3 *SOP for Data Review and Reporting*, SOP ID ADM-DATA_REV

21.4 *SOP for Nonconformance and Corrective Action*, SOP ID CE-QA008

21.5 *SOP for Handling Consumable Materials,* SOP ID ADM-CONSUM

21.6 *SOP for Training Policy,* SOP ID CE-QA007

21.7 *SOP for Laboratory Storage, Analysis, and Tracking,* SOP ID ADM-LabSAT

21.8 *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation,* SOP ID CE-QA011

21.9 *SOP for Manual Integration Policy,* SOP ID CE-QA002

21.10 *SOP for Evaluation & Pressurization of specially Prepared Stainless Steel Canisters,* SOP ID SMO-Can_Press

22) Appendix

22.1 Tables

Table 1 – Target Analytes with Corresponding Method Detection and Reporting Limits

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Data Review Checklist

TABLE 1

CARB Method 422 (Modified) Target Analytes with Method Reporting Limits

Analyte	MDL (ppb)	MRL (ppb)
Chloroform	0.095	1.0
Trichloroethene	0.042	1.0
Tetrachloroethene	0.061	0.20
1,2-Dibromoethane	0.18	0.50

Note: These values may change with each new MDL study performed. Additional compounds must have a complete MDL study and the MRL must be at or higher than the low standard of the initial calibration.

Attachment 1

Training Plan

Training Plan for Analysis of Various Halogenated Compounds by GC/ECD

Trainee _____ Trainer _____ Completion Date _____ Instrument ____

1. Read SOP — Trainer ____ Trainee ____ Date _____
2. Read Method: CARB 422 — Trainer ____ Trainee ____ Date _____
3. Demonstrated understanding of the scientific basis of the analysis
   Gas chromatography Electron Capture Detector — Trainer ____ Trainee ____ Date _____
4. Demonstrated familiarity with related SOPs — Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences; Rev.___
   SOP for Making Entries onto Analytical Records; Rev.___
   SOP for Manual Integration Policy; Rev.___
   SOP for Significant Figures; Rev.___
   SOP for Nonconformance and Corrective Action; Rev.___
   SOP for Performing MDL Studies and Establishing Limits of Detection & Quantitation; Rev.___
5. Observe performance of SOP — Trainer ____ Trainee ____ Date _____
   ___standard preparation (gas-phase dilutions)
   ___sample preparation
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant introduction
   ___data reduction and reporting
6. Perform SOP with supervision — Trainer ____ Trainee ____ Date _____
   ___standard preparation (gas-phase dilutions)
   ___sample preparation
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant use
   ___data reduction and reporting
7. Independent performance of the SOP — Trainer ____ Trainee ____ Date _____
   ___standard preparation (gas-phase dilutions)
   ___sample preparation
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant proficiency
   ___data reduction and reporting
   ___initial demonstration of competency
   ___ four consecutive laboratory control samples
8. Instrument operation and maintenance — Trainer ____ Trainee ____ Date _____
   ___GC and capillary column installation
   ___ECD setup and maintenance
   ___data system

Attachment 2
Initial Calibration Checklist

Initial Calibration Checklist
Analysis of Various Halogenated Compounds by Modified CARB 422

ICAL Date: _____

Instrument: ☐ GC21 ☐ _____

Analyst			Reviewer
☐	1.	Is the required documentation in the ICAL file?	☐
		☐ Sequence report	☐
		☐ Blank analysis Quantitation Report	☐
		☐ Calibration Status Report (aka Calibration History) - Initial	☐
		☐ Coefficient of Determination	☐
		☐ Quantitation Report for each calibration standard (including manual integration documentation – before and after printouts)	☐
		☐ ICV Quant Report and Evaluate Continuing Calibration Report (aka Percent Diff. report)	☐
☐	2.	ICAL performed continuously (i.e., not interrupted for maintenance or sample analysis)?	☐
☐	3.	ICAL performed within 48 hours?	☐
☐	4.	Standards analyzed from low concentration to high concentration?	☐
☐	5.	All analytes in blank analysis <MRL?	☐
☐	6.	Does each analyte's ICAL include a minimum of 5 concentrations?	☐
☐	7.	For each analyte, is there only one value used for each calibration level?	☐
☐	8.	If a point is dropped, is information noted in the ICAL explaining the reason?	☐
☐	9.	Does this follow the point dropping policy (including re-analysis within 48 hrs)?	☐
☐	10.	For each analyte, is the lowest standard's concentration at or below the MRL?	☐
☐	11.	For each analyte, does the ICAL include 5 consecutive levels?	☐
☐	12.	For each analyte, are there no levels skipped?	☐
☐	13.	Does the calibration curve give a correlation coefficient ≥0.98?	☐
☐	14.	For the ICV analysis, is the percent recovery for each analyte 70-130%?	☐
☐	15.	Are all peak integrations including manual integrations (per *SOP for Manual Integration Policy*) acceptable? ***If so, initial and date the appropriate pages***	☐

COMMENTS:

_____

_____

_____

Reviewed By _____ Secondary Reviewer _____

Date _____ Date _____

Attachment 3

Data Review Checklist

Data Review Checklist
Modified CARB 422

(Note exceptions in Comments section and attach Sample Preparation and Analysis Observations / Case Narrative Summary as applicable)

Analysis Date_____ Client _____ QC Level _____

Project # _____ Due Date _____ Instrument ☐ GC21 ☐ _____

Analyst **Reviewer**

Initial Calibration

☐ 1. Is the referenced ICAL the most recent ICAL performed? ☐

☐ 2. Has the referenced ICAL been peer reviewed and all associated documentation including the ICAL review checklist available for review? ☐

☐ 3. Were all associated requirements within the specified limits? ☐

Continuing Calibration

☐ 4. CCV raw data submitted? ☐

☐ 5. Was the %D for the CCV ±30% (first or second injection)? ☐

☐ 6. CCV analyzed at the beginning of the sequence, every 10 samples, and the end of the sequence? ☐

Sample Data

☐ 7. Is all sample data present and correct? ☐

- ☐ Sample raw data?
- ☐ Target analyte responses within calibration range?
- ☐ Peak integrations acceptable?
- ☐ All manual integrations flagged and properly documented? If so, initial and date.
- ☐ Any essential retention time shifts?
- ☐ All calculations correct?
- ☐ First quantitation report initialed and dated by analyst?

QC Data

☐ 8. Duplicate sample analyzed 1 per 20 or fewer samples? ☐

☐ 9. Is the laboratory duplicate within 15% of their average? ☐

☐ 10. Is the LCS/LCSD within ±15% of their average (where applicable)? ☐

☐ 11. Is the recovery for the LCS and/or LCSD within 70-130%? ☐

☐ 12. Are all analytes in the MB < MRL? ☐

Reporting Information

☐ 13. Sample Preparation and Analysis Observations / Case Narrative Summary completed if applicable? ☐

☐ 14. Appropriate flags indicated on a Sample Preparation and Analysis Observations / Case Narrative Summary form when applicable? ☐

☐ 15. Reporting spreadsheet complete and all flags correctly indicated? ☐

COMMENTS: _____

_____

_____

Reviewed By _____ Secondary Reviewer _____

Date _____ Date _____

ALS Standard Operating Procedure

Proprietary - Uncontrolled Copy

DOCUMENT TITLE:	DETERMINATION OF HYDROGEN, CARBON MONOXIDE, CARBON DIOXIDE, NITROGEN, METHANE, AND OXYGEN USING GAS CHROMATOGRAPHY WITH THERMAL CONDUCTIVITY DETECTION (TCD) IN ACCORDANCE WITH EPA METHOD 3C OR ASTM D 1946
REFERENCED METHOD:	EPA METHOD 3C, ASTM D 1946
SOP ID:	VOA-EPA3C
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STANDARD OPERATING PROCEDURE

DETERMINATION OF HYDROGEN, CARBON MONOXIDE, CARBON DIOXIDE, NITROGEN, METHANE, AND OXYGEN USING GAS CHROMATOGRAPHY WITH THERMAL CONDUCTIVITY DETECTION (TCD) IN ACCORDANCE WITH EPA METHOD 3C OR ASTM D 1946

EPA METHOD 3C, ASTM D 1946

SOP ID:	VOA-EPA3C	Rev. Number:	13.0	Effective Date:	12/31/2015

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DETERMINATION OF HYDROGEN, CARBON MONOXIDE, CARBON DIOXIDE, NITROGEN, METHANE, AND OXYGEN USING GAS CHROMATOGRAPHY WITH THERMAL CONDUCTIVITY DETECTION (TCD) IN ACCORDANCE WITH EPA METHOD 3C OR ASTM D 1946

1) Scope and Applicability

1.1 The referenced method (EPA Method 3C) was written for the analysis of carbon dioxide, methane, nitrogen and oxygen, in municipal solid-waste landfill gas and other stationary sources but is easily modified for the gas chromatographic method determination of hydrogen and carbon monoxide. In contrast, the practice ASTM D 1946 covers the determination of the chemical composition of reformed gases and similar gaseous mixtures containing each of these six components. Method ASTM D 1945-03 modified which describes the analysis of natural gas may also be referenced.

1.2 This method is appropriate for quantifying target analyte gases depending on the concentration of the samples from approximately 500 ppmv to high percent values. The number of samples, which may be analyzed in one eight hour day, is approximately twenty. The reporting limits for these analytes are listed in Attachment 4 of this standard operating procedure.

2) Summary of Procedure

2.1 The EPA Method 3C was written for use with backfilled summa canisters but is easily modified for samples collected as vapor in Tedlar bags, steel tanks, glass bottles, summa or other specially prepared canisters. In contrast, the ASTM methods do not specify a requirement for the sampling container.

2.2 An aliquot is drawn from the sampling container using a sample loop and injected onto a packed chromatographic column where the analytes are separated and measured using a thermal conductivity detector (TCD). Samples are analyzed in duplicate for EPA Method 3C, but a modification may be made which entails a single injection per submitted field sample. However, results from samples analyzed per ASTM D 1946 are obtained using a single injection technique.

Note: Refer to Sections 12.13 and 15.9 for the list of reporting modifications for these methods.

3) Definitions

3.1 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.2 Field Sample A sample collected and delivered to the laboratory for analysis.

3.3 Batch QC The QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) or Laboratory Duplicate (LD).

3.4 Calibration Standard (Initial Calibration – ICAL) A calibration standard of a known concentration containing desired analyte(s) prepared from a primary standard, which is, in turn, prepared from a stock standard material. A calibration standard is injected at varying volumes and used to calibrate the response of the measurement system with respect to analyte concentration.

3.5 Initial Calibration Verification (ICV) Standard An ICV is a standard that is obtained from a source other than the source for the calibration standards and is analyzed after the

measurement system is calibrated, but prior to sample analysis in order to verify the initial calibration of the measurement system.

3.6 Method Blank (MB) An analyte-free matrix, which is carried through the entire analytical process. It is used to evaluate the process for contamination from the laboratory.

3.7 Laboratory Control Sample (LCS) An LCS is a standard that is obtained from a source other than the source for the continuing calibration verification standard (CCV). The percent recovery of the analyte(s) in the LCS is used to assess method performance.

3.8 External Standard Calibration External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas or peak heights are compared to peak areas or peak heights of the standards.

3.9 Analytical Batch A group of samples which behave similarly with respect to the sampling or the test procedures being employed and are processed as a unit using the sample lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. In an analytical batch of samples, the time period is 24 hours or up to twenty sample injections, whichever comes first of continuous operation without interruption.

3.10 Continuing Calibration Verification (CCV) Standard A continuing calibration verification standard is a midrange calibration standard that is analyzed periodically to verify the continuing calibration of the measurement system.

3.11 Precision Precision of a method is how close results are to one another, and is usually expressed by measures such as standard deviation, which describe the spread of results.

3.12 Bias The bias of a method is an expression of how close the mean of a set of results (produced by the method) is to the true value.

3.13 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop “ticks” have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.14 Ambient Air Ambient air within the laboratory which is sampled and analyzed once per batch to assess injector performance.

3.15 Limit of Detection (LOD) The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DoD Clarification). For consistency purposes, the LOD may be referred to as the MDL once it is reported; however, full verification will be on file in the laboratory per the procedures detailed in this document.

3.16 Limit of Quantitation (LOQ) The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. (DoD Clarification). For consistency purposes and since the LOQ and MRL are equivalent with regards to laboratory procedure, the LOQ will be referred to as the MRL in this document and once it is reported. Full verification will be on file in the laboratory per the procedures detailed in the document.

3.17 Detection Limit (DL) / Method Detection Limit (MDL) The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type 1 error) is 1%. (DoD Clarification). For consistency purposes, the DL may be referred to as MDL. Also, as far as reporting is concerned the MDL will be raised (where necessary) to the verified LOD per the procedures defined in this document and reported accordingly.

4) Health and Safety Warnings

4.1 Each compound, mixture of compounds, standards, as well as samples, should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest level possible through the use of hoods (to minimize inhalation). For proper handling, use, and disposal refer to the laboratory's *Environmental Health and Safety Manual*, Safety Data Sheets (located in the safety cubicle in the front office), as well as the *SOP for Waste Disposal*.

4.2 Safety Data Sheets (SDS) Safety Data Sheets (SDS) are available in the Safety cubicle located in the front office and shall be reviewed as part of employee training.

4.3 Safety Glasses Safety glasses are required when performing maintenance on pressurized systems.

4.4 Pressurized Gases The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. The regulator should not remain on size "D" cylinders when not in use. Sources of flammable gases (i.e. pressurized hydrogen) should be clearly labeled.

4.5 Pollution Prevention and Waste Management All waste management must be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal* as well as the *Environmental Health and Safety Manual*.

5) Cautions

5.1 A maintenance log shall be kept documenting maintenance performed on each analytical system and the instrument maintenance log must be kept current and reviewed quarterly. The serial numbers of each instrument shall be recorded in the front of the logbook. An entry must be made in the appropriate log each time any maintenance activity is performed (no matter the extent). The entry in the log must include:

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful continuing calibration or initial calibration shall serve as proof that the maintenance is complete and the instrument is in working order.

5.2 Carrier Gas Purifier If in-line purifiers or scrubbers are in place, these purifiers must be changed as recommended by the supplier.

5.3 GC System

5.3.1 Column Column performance should be monitored by observing peak shapes and column bleed. Over time, the column may exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur depends on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be changed or the packing replaced (see Section 9.1.1). Care should be taken to minimize the introduction of air or oxygen into the column whenever GC maintenance is performed.

Decreasing performance can also be due to a leak in the system. Leaks can be detected with the use of a leak detector. Fittings may need to be tightened or ineffective column ferrules replaced to eliminate any leak detected.

5.3.2 Detector Replace filament assembly as needed.

5.3.3 Injection Lines Purge with nitrogen to ensure the line is not blocked.

6) Interferences

6.1 Contamination Dry ambient air at sea level contains 78.08% Nitrogen, 20.95% Oxygen, 0.93% Argon, and approximately 0.033% Carbon Dioxide by volume. Precautions must be taken to prevent intrusion of ambient air into the analytical system and the sampling containers.

6.1.1 Contamination in the Sample Care must be taken to prevent ambient air intrusion into the sample container during canister pressurization and laboratory analysis. When using adapters and fittings the dead volume should be evacuated and replaced with the sample gas prior to sampling from the container.

6.1.2 Carrier Gas Contamination To prevent system contamination, UHP/ZERO grade helium (99.999% purity) is used as the carrier gas. Also, a purifier and an oxygen trap are incorporated into the analytical system as additional insurance against possible contamination.

6.2 Peak Separation Since the TCD exhibits universal responses and detects all gas components except the carrier (helium, in this case), the appropriate temperature program, column flow rates and column packing must be used in order to separate all of the permanent gases with an exception of argon

6.3 Argon In this method, argon (0.93% by volume in ambient air) is not chromatographically separated from oxygen; therefore, results are reported as oxygen/argon.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review and reporting per the corresponding standard operating procedures. Laboratory personnel that have successfully demonstrated the ability to generate acceptable results according to this SOP are approved to perform sample analysis and interpretation of the results.

7.2 The department supervisor/manager or designee shall perform final review and sign-off on the data.

7.3 Demonstration of Capability

Training demonstrations shall be conducted in accordance with the *SOP for Training Policy,* DoD QSM 5.0, and TNI requirements. An initial demonstration of proficiency must be performed prior to independent analyses of samples. In addition, ongoing demonstration must be performed annually.

Once performance is found to be acceptable, a certification statement must be completed by the QA Manager and either the immediate supervisor or Laboratory Director and retained on file as a demonstration of compliance.

7.3.1 Quarterly Demonstration A demonstration of method sensitivity must be performed *quarterly on each instrument* performing this method.

1) A spike at the current LOD must be analyzed if results are to be reported below the MRL.
2) Verification of precision and bias at the LOQ must be performed.

Refer to Section 12.4 (LOQ) and 12.11.1 (LOD) for additional information on how these demonstrations are to be performed as well as the acceptance criteria.

7.3.2 Annual Demonstration Each analyst must perform this demonstration both initially and annually. Analyze four LCS standards at 1-4x the MRL (LOQ) either concurrently or over a period of days as a verification of precision and bias of the quantitation range. The standard deviation (n-1) and average percent

recovery of the four replicates are compared against current laboratory control limits for precision and bias. See Attachment 4.

7.3.3 Change in Personnel, Instruments, Method and/or Matrix The requirements in Sections 7.3.1 and 7.3.2 must be performed per the schedule noted and when there is a change in personnel, instruments, method or matrix. "Change" refers to any change in personnel, instrument, test method, or sample matrix that potentially affects the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, or other components of the sample analytical system, or a method revision).

All attempts at this demonstration must be completed and turned into the QA department for retention. Once performance is found to be acceptable, a required certification statement will be completed by the QA Manager and either the immediate supervisor or Laboratory Director and retained on file as a demonstration of compliance.

8) Sample Collection, Handling, and Preservation

8.1 The samples are collected and delivered to the laboratory for analysis in either Tedlar bags, specially prepared canisters, or glass sampling bottles (Bottle Vac. Entech Instruments). Samples collected in bags must be analyzed within 72 hours after sample collection unless otherwise specified by the client. Samples delivered in cleaned, evacuated summa or other specially prepared containers do not have a specified holding time for atmospheric gases but this laboratory recommends that samples be analyzed within 30 days from the date of collection.

9) Equipment and Supplies

9.1 Gas Chromatograph The analysis is performed using a Hewlett-Packard model 5890 series II gas chromatograph or equivalent equipped with a thermal conductivity detector.

9.1.1 Column 6' x 1/8" stainless steel column packed with 60/80-mesh carbosphere.

Conditioning of the chromatographic column is required prior to use of the system. The column should be conditioned with a continuous flow of chromatographic grade Helium and temperature programmed from 35˚C to 200˚C at a rate of five degrees per minute. The column should be held at 200˚C for at least four hours.

9.1.2 Sample Loop Stainless steel tubing with a 1/16" diameter (various lengths).

9.1.3 Conditioning System The system is able to maintain the column and sample loop at a constant temperature.

9.2 Adsorption Tubes In addition to a thermal gas purifier incorporated into the system, an oxygen trap shall be utilized to remove any O2 from the carrier gas to help in extending the life of the TCD filaments.

9.3 Sampling Media Tedlar bags, Summa canisters, or glass bottles may be supplied to the client for sampling purposes. These samples are submitted to the laboratory for analysis. Summa canisters must be conditioned and certified in accordance with the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters*.

10) Standards and Reagents

10.1 All samples, standards, and media must be stored separately. The concentration, preparation and expiration date as well as analyst's initials must be identified on the standard label. Each standard must also be uniquely identified with a laboratory ID number.

All standard certificates shall be noted with the standard identification number, date received and initials of the receiving analyst. They must then be given to the quality assurance department where they will be maintained. For additional information on these and other requirements, refer to the *SOP for Handling Consumable Materials.*

10.2 Carrier and Calibration Standard Balance Gas

10.2.1 Helium UHP/ZERO (99.999%) or higher in purity

10.3 Standards DoD compliance requires that second source standards be obtained from a second manufacturer. The use of a standard from a second lot is acceptable only when one manufacturer of the standard exists.

10.3.1 Purchased Standards These standards must be stored in accordance with the requirements described in the *SOP for Handling Consumable Materials*. These standards must be stored at ambient temperatures for a period of up to 2 years or as recommended by the manufacturer.

10.3.1.1 Scott Specialty Gas or Equivalent

Compound	Concentration
Carbon dioxide	~5.00%
Carbon monoxide	~5.00%
Hydrogen	~4.00%
Methane	~4.00%
Nitrogen	~5.00%
Oxygen	~5.00%
Balance Gas: Helium	

Note: The concentrations of these standards will change with each purchase and the specific concentration of each compound will be denoted on the standard as well as the Certificate of Analysis and used in all calculations.

10.3.1.2 Matheson or Equivalent

Compound	Concentration
Carbon dioxide	~5.00%
Carbon monoxide	~5.00%
Hydrogen	~4.00%
Methane	~4.00%
Nitrogen	~5.00%
Oxygen	~5.00%
Balance Gas: Helium	

Note: The concentrations of these standards will change with each purchase and the specific concentration of each compound will be denoted on the standard as well as the Certificate of Analysis and used in all calculations.

10.3.1.3 AirGas or equivalent (Neat gas standards)

Compound	Concentration
Hydrogen	99.999%
Oxygen	99.999%
Nitrogen	99.999%
Methane	99.999%
Carbon Dioxide	99.999%

10.3.2 Ambient Air Ambient air is analyzed once per batch to assess injector performance.

11) Method Calibration

11.1 Initial Calibration

Record the detector temperatures, GC temperature program, standard concentrations, and sample loop volume. All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, each analyte name, analyst's initials, concentration and response, response factor. Refer to Section 16.4 for the acceptance criteria.

11.1.1 Analysis Guidelines

- Analyze differing concentrations covering the desired calibration range by utilizing different sample loops. The dynamic range may be amended as long as all documentation reflects the correct concentrations.
- An ICAL shall be performed at a minimum annually.

11.1.2 Initial Calibration Requirements

Once a set of ICAL standards is analyzed, the previous ICAL may no longer be used to analyze new samples and it must be archived. The only time an archived ICAL can be used thereafter is to review or re-evaluate samples(s) previously processed using that ICAL.

1. A minimum of 5 concentrations, must be used to calculate the calibration curve.
2. Highest concentration, together with the lowest concentration, defines the calibration curve.
3. Lowest concentration must be at or below the method reporting limit.
4. The initial calibration event may not be interrupted by maintenance.
5. Only one value per concentration may be used.
6. Analyze calibration standards from low to high concentration.
7. All ICAL analyses must be completed within 48 hours.
8. One injection per 5 points (2 per 6) may be re-analyzed to replace "bad" injection(s).
9. Point dropping policy:
    - The following are guidelines to follow if points are to be reviewed to determine the appropriateness of dropping a point or injection.
    - Lowest concentration must be at the MRL and may not be dropped unless another concentration is added to the upper end of the curve. This would in turn raise the MRL.
    - Points at the high end may be dropped but another concentration must be added and used in the calculation. The curve range must be noted.
    - Points must not be dropped from the "interior" of a curve unless there is an assignable cause* for doing so that affects many (if not all) the analytes in the calibration standard. If a calibration standard

is to be dropped from the interior of the curve, all the analytes in the calibration standard must be dropped from all the analytes' calibration curves.

- If a point or a calibration standard is dropped, the reason must be documented (and the results maintained with the documentation for the final ICAL).
- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 48 hours).
- Once the ICAL has been used to calculate and report sample results, it is not to be changed.

* Assignable causes include

- Standard preparation error
- Instrument malfunction (e.g., it quits acquiring in the middle of the analysis)
- Bad injection or purge

10. A set of concentrations for a calibration curve is in the following table (Attachment 5). However these concentrations might change due to the availability of the standards. Other concentrations can be used as long as all other guidelines for the analysis of initial calibration are followed.

Note: Hydrogen may not be linear; therefore, if an average response factor or linear regression cannot be used, a quadratic curve fit may be employed. A quadratic (second order) model requires a minimum of five calibration points.

11.1.3 ICAL Update Procedure

1. Open most recent method.
2. Save to new ICAL method ID. The date used in method ID is the date files were analyzed.
3. Clear all responses prior to update initiation and/or clear levels if different concentrations are to be used (Initial Calibration → Clear All Calibration Responses; Initial Calibration → Clear All Calibration Levels).
4. Quantitate standard
5. Review all peaks for retention time, integration, etc.
6. Update responses for standard
7. Repeat for all standards
8. If necessary load midpoint standard and update retention times.
9. Save method.
10. Verify Calibration Files listed on Response Factor Report are correct (Both Primary and Secondary Reviewer).
11. Verify responses of Page 3 of Edit Compounds are correct (Both Primary and Secondary Reviewer).
12. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report (Both Primary and Secondary Reviewer).
13. Save Method. Confirm that no other copies of the method are open on other computer workstations.

Note: It is also acceptable to quantitate all standards and review all peaks before updating responses but steps 1-2 still must be completed initially. Step 3 also must be done prior to beginning ICAL update.

11.1.4 Initial Calibration Review

The ICAL checklist is used to document the review and approval process. The Analyst's calculation and assessment along with a peer review of all ICAL data

and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples.

11.1.5 Initial Calibration File

An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- Blank analysis quantitation report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Plot for quadratic fit for hydrogen, if necessary
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and evaluate continuing calibration report (aka Percent Difference Report)
- Injection log (optional)

11.1.6 Initial Calibration Verification Verify the initial calibration by analyzing an independent calibration verification standard (ICV). Utilize the standard described in Section 10.3.1 for the analysis of a second source standard. Refer to Section 16.5 for acceptance criteria.

12) Sample Preparation/Analysis

12.1 Analytical Sequence The analytical batch must be completed for the analysis of ≤20 field samples.

Analytical Sequence Guideline[1]

Sample Description (w/ICAL)	*Sample Description*
Calibration Stds.[2]	CCV[3]
ICV[4]	MB[5]
MB[5]	Lab Air[6]
Lab Air[6]	Samples 1-10[7]
Samples 1-10[7]	CCV[3]
CCV[3]	Samples 11-19[7]
Samples 11-19[7]	LD[8]
LD[8]	LCS[9]
CCV[3]	CCV[3]

[1]The batch QC may be analyzed in an order other than the one listed in this document; the analytical sequence specified below is a guideline.
[2]The initial calibration must be generated in accordance with the guidelines detailed in Section 11.1.1 of this document.
[3]In cases, where the ICAL is not performed the analytical sequence must begin with the analysis of a CCV standard. In an external standard calibration the CCV is to be analyzed no less frequently than every ten samples or every 12 hours, whichever is more frequent, and the analytical sequence is to end with the analysis of a CCV standard.
[4]Every ICAL must be followed by a second source standard (ICV) which contains all of the target analytes. Same source as LCS; therefore, LCS is not required to be analyzed again.
[5]The method blank must be carried throughout the entire analytical process and be analyzed prior to any samples within the sequence. A method blank (MB) shall be run to monitor for laboratory introduced contamination.
[6]A volume of laboratory ambient air shall be analyzed at a rate of one per twenty sample injections or fewer.

[7]EPA Method 3C requires a duplicate injection for each sample. If the samples are being analyzed per a modified Method 3C, they are to be injected once (refer to note number 8). ASTM D 1946 requires only a single injection.

[8]Every batch must include the analysis of a laboratory duplicate. Samples selected for duplicate analysis shall be rotated among client samples. In addition, if performing EPA Method 3C without modification (duplicate injection), the laboratory duplicate analysis will not be necessary. A laboratory duplicate is considered a sample.

[9] A second source standard similar to 10.3.1.1 shall be analyzed once per twenty sample injections or fewer.

12.2 Conditions

The column and detector temperatures should be adjusted to the recommended levels. The column should be conditioned as instructed in Section 9.1.1. Once the GC/TCD system is optimized for analytical separation and sensitivity, the identical sample operating conditions must be used to analyze all samples, blanks, calibration standards and quality control samples.

The recommended settings and system parameters for GC01 are as follows:

Sample Inlet: GC
Injection Source: Sample Loop
Run Time: ~8 min

OVEN

Initial Temperature: 50˚C *Maximum Temperature*: 250˚C
Initial Time: 2.0 min *Equilibration Time*: 0.0 min

Ramps: Rate: 30˚/min
Final Temp.: 200˚C
Final Time: 1 min

COLUMN

Type: Packed
Model: Carbosphere 60/80
Dimensions: 6’ x 1/8”

DETECTOR

Temperature: 260˚C
Reference Flow: 45mL/min
He Make up: 20mL/min

The recommended settings and system parameters for GC20 are as follows:

Sample Inlet: GC
Injection Source: Sample Loop
Run Time: ~6.5 min

OVEN

Initial Temperature: 50˚C *Maximum Temperature*: 250˚C
Initial Time: 1.0 min *Equilibration Time*: 0.0 min

Ramps: Rate: 30˚/min
Final Temp.: 200˚C
Final Time: 0.5 min

COLUMN

Type: Packed
Model: shin carbon ST 100/120
Dimensions: 2 meters 1mm ID

DETECTOR

Temperature: 300˚C
Reference Flow: 20mL/min
He Make up: 2mL/min

12.3 Retention Time (RT) Windows

Retention time windows for each target analyte must be generated whenever there is a major change in instrument conditions including flow rates or when standard analyses result in analyte retention times outside the established windows. The procedure for determining the retention time windows for this method is as follows. However, other approaches may be employed, providing that the analyst can demonstrate that they provide performance appropriate for the intended application. For example, the analyst may use the corresponding retention times from the initial calibration as they may show shifts in RTs due to the volume injected (higher concentrations lead to wider peaks).

1. Make sure that the system is operating reliably and that the system conditions have been optimized for the target analytes in the sample matrix to be analyzed.
2. Make four injections of all applicable standard mixes over a 72 hour period. Make the injections cover the entire 72-hour period or the end result could be windows, which are too tight.
3. Record the retention time for each single component analyte to three decimal places. Calculate the mean and standard deviation of the four absolute retention times for each single component analyte and surrogate
4. If the standard deviation of the retention times for the target compound is 0.000, then additional injections may be included or the use of a default standard deviation of 0.01 minutes.
5. The width of the retention time window for each analyte is defined as ±3 times the standard deviation of the mean absolute retention time established during the 72 hour period. If the default standard deviation of 0.01 is used, the width of the window will be 0.03 minutes.
6. Establish the center of the retention time window for each analyte by using the absolute retention time for each analyte from the continuing calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Retention time windows must be calculated for each analyte on each instrument. New retention time windows must be established when a new column is installed.

12.4 LOQ Establishment, Verification, and Acceptance Criteria

A) The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
B) The LOQ for each analyte must be ≥ the analyte's LOD.
C) Initially a passing demonstration of precision and bias must be performed at the LOQ.
D) Run CCV 2 times at LOQ and:
   1) Evaluate the LOQ for precision and bias using current control chart limits.
   2) Check the signal to noise ratio (S/N) using the software. The S/N ratio must be at least 3:1 for each analyte.
E) If anything fails, verify at higher level and notify reporting. Also, make a note in the ICAL documentation.
F) Turn in all LOQ verification data (quant reports and software reports/checks) to QA (regardless of pass/fail).
G) Verify the LOQ on each instrument quarterly by running the CCV at the LOQ and verifying that ongoing precision and bias requirements are met.

12.5 Continuing Calibration Verification

A continuing calibration check shall be performed at the beginning and end of an analytical sequence and every ten field samples, not to exceed a 12 hour period. The concentration of the calibration verification may be varied within the established calibration range. Refer to Section 16.6 for acceptance criteria.

12.6 Laboratory Control Sample

A second source standard similar to Section 10.3.1.1 shall be analyzed once per closed batch. Refer to Section 16.11 for acceptance criteria.

12.7 Method Blank

A method blank must be analyzed by sampling chromatographic grade helium. Refer to Section 16.8 for acceptance criteria.

12.8 Sample Analysis

Refer to Section 16.10 for the acceptance criteria.

12.8.1 Container Pressurization Sample analysis must be made using the same instrument parameters as that of the calibration standards. Refer to the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters* for the procedure of how containers are to be pressurized prior to analysis. The analyst shall record the appropriate pressures on the Service Request form.

12.8.2 Sample Analysis Sample analysis is performed with the utilization of a sample loop equipped with a pump. If the sample container is not equipped with a sampling valve appropriate for this use, the sample container shall be fitted with an adapter. The dead volume within the adapter shall be evacuated and the sample loop flushed then filled with sample gas. Analyze each sample in duplicate (calculate the percent difference of the calculated concentration of each analysis) unless performing a single injection modification or referencing ASTM D 1946 (refer to Section 12.8.3, #2).

Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments). Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fitting higher than 80ºC.

12.8.3 Sample Re-analysis

1. If the response of any permanent gas analyte in a sample is greater than the response of that analyte in the ICAL (outside the ICAL upper calibration range) the sample shall be reanalyzed using a smaller loop.

   Dilution (i.e. Tedlar bags) would compromise sample integrity with the addition of laboratory air. Guidance in performing dilutions and exceptions to this requirement are given below.

   - The dilution factor chosen should keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument. Additional compounds may be reported as long as they are within the calibration range.

2. If the percent difference between the duplicate injection (analysis without modification) is greater than the acceptance criterion of 5%, the sample must be re-analyzed and repeated until acceptable *consecutive* numbers are achieved.

12.9 Laboratory Duplicate (LD)

If the method is being performed with a single injection modification, then the analysis of a LD is required to show precision. The laboratory duplicate should be rotated among clients, whenever possible. Refer to Section 16.9 for acceptance criteria.

12.10 Manual Integration

The integration for each peak is checked to ensure that it has been integrated properly. Assuming an incorrect automatic integration the analyst shall conduct the manual integration in accordance with the *SOP for Manual Integration Policy* including all documentation and reviews associated with the process. The review shall include the analyst and peer reviewer initialing and dating the manual integration as an indication of acceptability and approval.

12.11 Detection Limits and Limits of Detection

If results are to be reported below the MRL, an MDL study must be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* Method detection limits must be determined annually and each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation is such that it affects the sensitivity of the analysis. The MDL study shall be performed on each instrument for which this method is performed. All supporting data must be approved and retained.

The detection limit shall be used to determine the LOD for each analyte. Once determined on each instrument, the highest LOD (for each analyte from all instrument determinations) shall be used as the uniform LOD.

12.11.1 Performance and Acceptance Criteria

1. Perform Limit of Detection (LOD) verification on all instruments (performing this method) immediately following the MDL study. Spike the LOD at 2-4x the MDL; the spike level establishes the LOD.
2. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).
   - It is specific to each combination of analyte, matrix, method and instrument configuration.
   - The LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.
3. If the LOD verification fails (per #2), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.
4. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

Note: Per the DoD QSM 5.0 and TNI Standard, it is not necessary to perform a MDL study when results are not to be reported below the LOQ/MRL.

12.12 Ambient Air

An ambient laboratory air sample shall be analyzed once per closed batch (20 or fewer sample injections). Refer to Section 16.7 for the acceptance criteria and corrective action.

12.13 Method Modifications

12.13.1 The following are EPA 3C method modifications:

- Reporting carbon dioxide, methane, nitrogen, and oxygen from a single sample injection.
- Reporting hydrogen and carbon monoxide (these compounds are not included in 3C method).
- Sample results are normalized per ASTM D 1946.
- Use of sample containers other than backfilled Summa canisters.

12.13.2 The modification for ASTM D 1946 is the omission of ethane and ethane.

12.13.3 The column backflush procedure described in method ASTM D 1945-03 is not performed.

12.14 Loop calibration

The loop injection port has a standard loop of approximately 100ul to introduce sample to the instrument. There are other loops that are used to introduce smaller and larger amounts and these are calibrated against the normal loop for a known dilution factor.

12.14.1 Calibration Procedure

A standard of approximately 50000ppm for all analytes is analyzed three times with the normal loop. The area counts for all analytes with the exception of hydrogen are summed for each standard. This summation is averaged of the three standard injections. This procedure is duplicated using another loop. The dilution factor is the ratio of the average area counts of the normal loop divided by the average area counts of the other sampling loop.

For current Loop Ratios see Table 1.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Data System

Load the appropriate analytical sequence (e.g., J:\GC1\sequence\fxgs_25c.s). Enter the analytical sequence information in the table window, including sample/standard name. Load the appropriate quantitation analytical method (e.g., J:\gc1\methods\"appropriate ICAL"). Run the sequence and analyze the standards and samples in the order specified.

14.2 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. Files shall be named with a two-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files shall be saved in a unique sub-directory on the server. An example of how the analyst must store analytical data is as follows:

Instrument Number/Data/Method ID/yr_month/*.d

* Injection (automatically assigned based on order of injection)

14.3 Sufficient raw data records must be retained of the analysis, instrument calibrations and method detection limit studies. This includes analysis/calibration date, test method, instrument, sample identification, each analyte name, analyst's initials, concentration and response, and standards used for the analysis and calibrations as well as any manual integrations and all manual calculations including sample dilutions. All information entered and reported on the quantitation reports must be complete and accurate.

14.4 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time of analysis (both are required for Tedlar bags since the holding time is 72 hours), instrument operating conditions/parameters (or reference to such data), analysis type, manual integrations, all manual calculations, analyst's initials, sample preparation (pressure readings and balance gas), standard and reagent origin, sample receipt, calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, and assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 Initial Calibration

- Response Factor for each injection (equation number 5)
- Mean Response Factor using all injections (equation number 6)
- Percent Relative Standard Deviation (equation numbers 5,6,7, and 8)

Hydrogen (if quadractic is used):

- Coefficient of Determination (equation number 12)

15.2 Initial Calibration Verification

- Response Factor (equation number 5)
- Mean Area Response (equation number 6)
- Percent Difference (equation number 3)

15.3 Continuing Calibration Verification

- Response Factor (equation number 5)
- Mean Area Response, where necessary (equation number 6)
- Percent Difference (equation number 3)

15.4 Laboratory Duplicate and Method 3C without modification

- Relative Percent Difference (equation number 4)

15.5 Sample Analysis

Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.

All permanent gas results are normalized as dry gas to 99.99% proportionately, in order to reflect the true composition of the sample. It is the practice of the laboratory to normalize results of permanent gas analysis, except under special circumstances that occur where the normalization of the results is not utilized or the normalization procedure is modified. For example, samples containing greater than 0.01% by volume of measured constituents other than permanent gases (for instance high hydrocarbon or sulfur levels) are normalized to 99.99% minus the percent contribution from components other than permanent gases.

- Calculate the average area of the two injections, where necessary (equation number 2)
- Calculate the dilution factor, where necessary (equation number 1)
- Analyte concentration (equation number 9)
- Hydrogen concentration (equation number 14)
- Normalization (equation number 11)

When the analysis of a sample produces permanent gas results whereby the total is significantly less than expected, accounting for experimental error, it is the laboratory's practice to reanalyze the sample in question as well as the laboratory air. This will determine if there is a problem with the analytical system. If there is no problem with the system and the results are the same refer to the following example.

If the total of the permanent gas analysis is less than 60.0% by volume and the laboratory is not requested to perform additional analyses, the results would be reported unnormalized. The decisions whether to report the unnormalized results is at the discretion of the analyst and department supervisor.

15.6 Laboratory Control Sample

- Calculate the percent recovery (equation number 10)

15.7 Calculations

15.7.1 Equation Number 1

Dilution Factor

$$DF = \frac{V_{STD}}{V_S}$$

Where:

DF = dilution factor
V_{STD} = volume of standard loop
V_S = volume of sample loop

15.7.2 Equation Number 2

Average

$$\frac{x + y}{n}$$

where:

x = response from the first injection
y = response from the second consecutive injection
n = number being averaged together

15.7.3 Equation Number 3

Percent Difference, %D,

The %D is used for evaluating ICV and CCV vs. the initial calibration

$$\%D = \frac{C_{CCVorICV} - C_{std}}{C_{std}}(100)$$

where, for any given analyte:

$C_{CCVorICV}$ is the calculated concentration being evaluated
C_{std} is the concentration of the standard used

15.7.4 Equation Number 4

Relative Percent Difference (RPD)

$$\frac{|R_1 - R_2|}{\left(\frac{R_1 + R_2}{2}\right)} x100$$

where:

R_1 First measurement value
R_2 Second measurement value

15.7.5 Equation Number 5

Response Factor (RF)

The response factor, for analyte *x* is given by:

$$RF = \frac{A_x}{C_x}$$

where:

A_x = Area of the analyte in the standard
C_x = Concentration of the analyte in the standard

15.7.6 Equation Number 6

Average (or Mean) RF

$$\overline{RF} = \frac{\sum_{i=1}^{N} RF_i}{N}$$

where:
RF_i are the individual RFs from each injection in the initial calibration curve
N is the number of injections

15.7.7 Equation Number 7

Standard Deviation, SD:

$$SD = \sqrt{\sum_{i=1}^{N} \frac{\left(RF_i - \overline{RF}\right)^2}{N-1}}$$

where:

RF_i are the individual RFs from each concentration level in the initial calibration curve

$\overline{RF}$ Average (or Mean) RF of all injections in the initial calibration curve

N total number of injections

15.7.8 Equation Number 8

Percent Relative Standard Deviation, %RSD:

$$\%RSD = \frac{SD}{\overline{RF}}(100)$$

where:

SD Standard Deviation calculated in equation number 3

$\overline{RF}$ Average or Mean RF

15.7.9 Equation Number 9

Concentration (C):

$$C = \frac{Area}{\overline{RF}} \times \frac{D_{SLV}}{A_{SLV}}$$

or

$$C = \frac{\overline{Area}}{\overline{RF}} \times \frac{D_{SLV}}{A_{SLV}}$$

where:

Area is the area obtained from the chromatogram

$\overline{Area}$ Mean area for both injections, if performing analysis without modification

$\overline{RF}$ Average (or Mean) RF of all concentration levels in the initial calibration curve

D_{SLV} default sample loop volume

A_{SLV} actual sample loop volume

15.7.10 Equation Number 10

Percent Recovery (%R):

$$\%R = \frac{C}{S} x100$$

where:

C = Concentration of the analyte recovered
S = Spiked amount

15.7.11 Equation Number 11

Normalization
Divide each analyte's calculated concentration (percent) by the percent sum of the permanent gases in the sample and multiply by 99.99 or the adjusted value.

15.7.12 Equation Number 12

Quadratic (Coefficient of Determination)

$$COD = \frac{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right)\sum_{i=1}^{n}(y_{obs} - Y_i)^2}{\sum_{i=1}^{n}\left(y_{obs} - \bar{y}\right)^2}$$

where:

y_{obs} = Observed response (area) for each concentration from each initial calibration standard

$\bar{y}$ = Mean observed response from the initial calibration

Y_i = Calculated response at each concentration from the initial calibration

n = Total number of injections

p = Number of adjustable parameters in the polynomial equation (i.e., 3 for a third order; 2 for a second order polynomial)

15.7.13 Equation Number 13

Quadratic Fit

$R = AX^2 + BX + C$

where:

R = response
X = quantity, ng
A, B and C = are coefficients in the equation

15.7.14 Equation Number 14

Analyte Concentration (using equation number 13)

$$X = \frac{\sqrt{4A(R-C) + B^2} - B}{2A}$$

15.8 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated following the data review checklist in Attachment 3. The

data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The data review checklist shall be used to document the review process. Once it has been completed, the checklist must be initialed, dated and filed with each job file. Results must not be reported until after they are appropriately reviewed according to this SOP, the *SOP for Data Review and Reporting* and the *SOP for Laboratory Ethics and Data Integrity*.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.5.

15.9 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and all information required by this SOP and the *SOP for Data Review and Reporting*. The following are situations whereby the results shall be reported as being analyzed by Modified EPA Method 3C: single injection, reporting hydrogen and carbon monoxide and if analyzing replicate injections (for 3C without modification) and the samples are submitted in Tedlar bags.

15.9.1 EPA Method 3C Modifications

- Single injection
- Sample container other than backfilled Summa canisters
- Reporting carbon monoxide and /or hydrogen

15.10 Sample Preparation and Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis, and Tracking* must be generated when there are any specific sample composition information, sample preparation, analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved.

This form is necessary as a means for documenting any unusual or noncompliant information. This form, among other information, will be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through preparation, analysis, data reduction, and reporting.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 This section of the standard operating procedure contains technical acceptance criteria and preferred corrective actions to data nonconformities. Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate.

16.2 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.3 It must be determined if there are any instrumentation problems contributing to out of control QC data and the analyst must determine if this has affected sample results. This being the case, all samples (including QC) that are affected by instrumentation problems must be re-analyzed following any necessary maintenance activity.

16.4 Initial Calibration

16.4.1 Acceptance Criteria

- If a quadratic fit (for hydrogen) is used it should be forced through zero.
- The percent relative standard deviation (%RSD) for the response factors must be ≤15% for all compounds except hydrogen if utilizing a quadratic curve.
- Hydrogen may be fitted to a quadratic curve where the coefficient of determination (COD) shall be ≥0.99.
- The retention time for each point must within 0.06 minutes of the mean RT. However it must be noted that higher injection volumes and/or higher concentrations of any analyte may not meet this criteria, which is acceptable.

16.4.2 Corrective Action

If the initial calibration technical acceptance criteria are not met, inspect the system for possible sources. Check standards and re-analyze (per ICAL policy in Section 11.1.2), if necessary. Also, it may be necessary to perform maintenance or perform other corrective actions to meet the technical acceptance criteria. Attempt another initial calibration and make a notation in the maintenance logbook regarding any maintenance steps taken. If the recalibration does not meet the established criteria, new calibration standards must be made. A demonstration of an in-control system is required before proceeding with the analysis.

16.5 Initial Calibration Verification (ICV) Standard

16.5.1 Acceptance Criteria

- The percent difference for each compound in the ICV must be ≤15%.

16.5.2 Corrective Action

If the ICV does not pass the criteria the standard must be reanalyzed and reevaluated. If reanalysis also fails to produce an acceptable recovery, documented corrective action must be initiated. This may include instrument maintenance, a new ICV standard or the analysis of a new initial calibration curve.

16.6 Continuing Calibration Verification (CCV) Standard

16.6.1 Acceptance Criteria

- The percent difference for each analyte in the CCV must be ≤10%, except hydrogen which must be ≤15%.
- The retention time for each analyte in the standard must be within 0.33minutes of the mean RT (of the corresponding analyte) from the ICAL.

16.6.2 Corrective Action

If the continuing calibration fails to meet expected criterion, the CCV may be reanalyzed (no more than two runs of the CCV standard may be analyzed without documented corrective action, i.e. a notation in the logbook). If the acceptance criterion is still not met, it may be necessary to perform maintenance prior to reanalysis. If routine maintenance does not correct the problem, a new initial calibration must be performed on the instrument.

If the retention time criterion is not met, leak check the system, check the carrier gas cylinders, determine if there has been a loss of pressure in lines. If the analytes do not fall within the generated windows, a new retention time window should be generated.

DoD QSM 5.0 Requirement: If a CCV fails, the laboratory must immediately analyze two additional consecutive CCVs (immediately is defined as within one hour).

- Both of these CCVs must meet acceptance criteria in order for samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

16.7 Ambient Air

16.7.1 Acceptance Criteria

- The sum of the results for nitrogen and oxygen/argon must fall between 90% and 110% (un-normalized).

16.7.2 Corrective Action

Reanalyze the lab ambient air and if the results still do not meet the criterion, the sample line should be purged with nitrogen to release any blockage. This is particularly important if the results for the first criterion are low. Also, if the result is low the system should be checked for leaks. All standards, samples and QC samples associated with the lab ambient air should be reanalyzed following the maintenance activity if it is determined that the results could have been affected.

16.8 Method Blank

16.8.1 Acceptance Criteria

- The method blank result for any target analyte must not be greater than the method reporting limit. Also, the blank should not contain additional compounds with elution characteristics that would interfere with identification and measurement of a target analyte.
- For DoD samples, the method blank will be considered to be contaminated if:
  1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
  2. The concentration of any common laboratory contaminant in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
  3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

  The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

16.8.2 Corrective Action

Re-inject the method blank and if the results are the same, analyze an instrument blank (inject without turning on the pump) to determine if the contamination is the blank canister or the analytical system. Corrective action documentation must be initiated following a failed second analysis. If the

system is contaminated, then both the method blank(s) and the associated samples in question must be re-analyzed.

16.9 Laboratory Duplicates (Modified EPA Method 3C)

16.9.1 Acceptance Criteria

- Every batch of twenty or fewer samples, if performing EPA Method 3C with modification, must include the analysis of a laboratory duplicate as a measurement of method precision. Refer to Attachment 4 of this document.

16.9.2 Corrective Action

If the replicate results do not fall within the technical acceptance window, the sample should be re-analyzed. If the results are still unacceptable and there does not appear to be any matrix effects, interfering peaks, or instrument problems, the results for both injections shall be reported to the client with the appropriate qualifier.

16.10 Sample Analysis

16.10.1 Acceptance Criteria

- Samples out of holding time must be handled according to Section 16.12.
- The sample replicate injections are acceptable when the RPD is within ±5% (analysis without modification must consist of consecutive injections).
- Analyte retention time must be within the daily RT window and within 0.33minutes of the mean RT in the ICAL.

16.10.2 Corrective Action

Analysis Without Modification If the two injections do not agree, run additional samples until consistent area data are obtained in two consecutive injections.

Analysis With or Without Modification If the retention time for any analyte falls outside of the retention time window from the latest daily calibration or average initial calibration retention time, the system must be inspected for a change in the head pressure and the results evaluated and reported accordingly.

Results not bracketed by initial instrument calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags.

16.11 Laboratory Control Sample (LCS)

16.11.1 Acceptance Criteria

- The percent recovery must fall within the fixed recoveries of 85-115% or laboratory generated control limits when available. Refer to Attachment D.

16.11.2 Corrective Action

If the LCS criteria are not met, determine whether the cause is instrumentation problems, result of poor injection or a poor LCS. If necessary perform maintenance, re-inject the LCS or make a new standard. If the LCS criteria are still not met, a new ICAL must be run or the data must be qualified.

16.12 Expired Sample Holding Time

The customer shall be notified by the Project Manager (best attempt) when informed by an Analyst, Team Lead or SMO that the sample's holding time was missed. The customer must decide if the sample analysis shall continue. The documentation of missed holding time and the client's decision to proceed must be included in the

corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s) as detailed in Appendix D of the most current Quality Assurance Manual.

18.2 When analysis quality control results are unacceptable:

If the associated samples are within holding time, re-analyze the sample with criteria under control. Alternatively, evaluate the effect on the sample results and report the results with qualifiers and/or discuss in the case narrative if the effect is judged insignificant.

18.2.1 Method Blank If an analyte in the method blank is found to be unacceptable and the analyte is also found in associated samples, those sample results shall be "flagged" in the report. If the analyte is found in the blank but not in the sample and all other quality control meets acceptance criteria then the results for the sample may be reported without a qualifier. However, if other QC is out of control then an evaluation must be made and the results reported accordingly.

18.2.2 Laboratory Duplicate (Analysis with Modification) If the results from the reanalysis are unacceptable, and there does not appear to be any matrix effects, interfering peaks, or instrument problems, the results for both injections shall be reported to the client. In addition, other results from the same analytical sequence should be reported with the appropriate qualifier.

18.2.3 Laboratory Control Sample An unacceptable LCS must be evaluated along with the sample analysis and reported accordingly.

18.2.4 Initial Calibration Sample data may NOT be reported with an unacceptable ICAL.

18.2.5 CCV Sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special condition:

When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the sample affected by the unacceptable CCV shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

18.3 Sample Out of Control

18.3.1 Hold Time All Tedlar bag samples analyzed outside of the required hold time of 72 hours must be reported with the appropriate qualifier.

18.3.2 Retention Time All analytes outside of the retention time window (following a retention time evaluation) must be reported with the appropriate qualitative uncertainty, where necessary.

18.3.3 Duplicate Results (Analysis without modification) If the results from any of the repeated injections are still unacceptable (and other sample results were acceptable), and there does not appear to be any matrix effects, interfering peaks, or instrument problems, the results for both injections shall be reported to the client. If the out-of-control results are due to matrix interferences, report the results with a matrix interference qualifier.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

19.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. MDLs can be obtained using standards at a concentration of about 300ppm to 1000ppm and making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence.

The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. Refer to Section 12.11.1 for the LOD verification criteria.

Note: Per the DoD QSM 5.0 and TNI Standard, it is not necessary to perform a MDL study when results are not to be reported below the LOQ/MRL.

19.3 Accuracy and Precision

Refer to Section 16.9 for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 15%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in attachment 4. Refer to Section 12.4 for the accuracy and precision LOQ requirements.

19.4 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Ongoing demonstration of capability shall be performed and documented; however, the initial demonstration of method capability is not required.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
13.0	12/31/15	C. Humphrey	7.3 – Removed reference to NELAC
			12.11.1 – Removed reference to NELAC
			12.13.1 – Revised to add clarification to EPA 3C method modifications
			19.2 – Removed reference to NELAC
			Attachment 4 – Updated control limits

21) References and Related Documents

21.1 "*Determination of Carbon Dioxide, Methane, Nitrogen, and Oxygen from Stationary Sources*", EPA Method 3C

21.2 ASTM D 1946-90 (Reapproved 2006), "*Standard Practice for Analysis of Reformed Gas by Gas Chromatography*".

21.3 ASTM D 1945-03 (Reapproved 2010), "*Standard Test Method for Analysis of Natural Gas by Gas Chromatography".*

21.4 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.

21.5 *SOP for Batches and Sequences*, SOP ID ADM-BATCH_SEQ

21.6 *SOP for Making Entries onto Analytical Records*, SOP ID CE-QA007

21.7 *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*, SOP ID CE-QA011

21.8 *SOP for Manual Integration Policy*, SOP ID CE-QA002

21.9 *SOP for Nonconformance and Corrective Action*, SOP ID CE-QA008

22) Appendix

22.1 Tables

Table 1 – Loop Ratios

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Data Review Checklist

Attachment 4 – MRLs and Control Limits

Attachment 5 – Calibration Curve Concentrations

Table 1

Loop Ratios	
Normal Loop	1.00
Small Loop	0.1556
Medium Loop 1	0.4202
Medium Loop 2	0.8521
Large Loop	1.280

Note: New loop ratios may be established prior to the revision of this document, refer to the most recent loop ratios.

Attachment 1

Training Plan

Training Plan for Analysis of Fixed Gases by GC/TCD

Trainee _____ Trainer _____ Instrument _____

1. Read SOP Trainer ____ Trainee ____ Date _____
2. Read Method: EPA Method 3C, ASTM D 1946, ASTM D 1945 Trainer ____ Trainee ____ Date _____
3. Demonstrated understanding of the scientific basis of the analysis
   Gas chromatography Thermal Conductivity Detector Trainer ____ Trainee ____ Date _____
4. Demonstrated familiarity with related SOPs Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences
   SOP for Making Entries onto Analytical Records
   SOP for Manual Integration Policy
   SOP for Significant Figures
   SOP for Nonconformance and Corrective Action
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation
5. Observe performance of SOP Trainer ____ Trainee ____ Date _____
   ___standard preparation
   ___sample preparation (gas-phase dilutions)
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant introduction
   ___data reduction and reporting
6. Perform SOP with supervision Trainer ____ Trainee ____ Date _____
   ___standard preparation
   ___sample preparation (gas-phase dilutions)
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant use
   ___data reduction and reporting
7. Independent performance of the SOP Trainer ____ Trainee ____ Date _____
   ___standard preparation
   ___sample preparation (gas-phase dilutions)
   ___analytical sequence setup
   ___initial calibration and continuing calibration verification
   ___sample analysis
   ___EnviroQuant proficiency
   ___data reduction and reporting
   ___initial demonstration of competency
      ___ Four consecutive laboratory control samples
8. Instrument operation and maintenance Trainer ____ Trainee ____ Date _____
   ___gas chromatograph and column installation (packed)
   ___detector (TCD) setup and maintenance
   ___data system

Attachment 2

Initial Calibration Checklist

Initial Calibration Checklist (Fixed Gases)

Analysis: EPA Method 3C / ASTM D 1946 / ASTM D 1945

ICAL Date _____ Instrument ☐ GC01 ☐ GC ____

Analyst		Reviewer
☐	1. Is the required documentation in the ICAL file?	☐
	☐ Sequence report	
	☐ Blank analysis Quantitation Report	
	☐ Calibration Status Report (aka Calibration History) – Initial	
	☐ Response Factor Report and Plot for Hydrogen	
	☐ Quantitation Report for each calibration standard (including manual integration documentation – before and after printouts)	
	☐ ICV Quantitation Report and Evaluate Continuing Calibration Report (aka Percent Diff. report)	
☐	2. Was the ICAL performed continuously (i.e., not interrupted for maintenance or sample analysis)?	☐
☐	3. All the calibration standards analyzed within 48 hours of each other?	☐
☐	4. Were the standards analyzed from low concentration to high concentration?	☐
☐	5. Are all the analytes in the blank analysis < MRL?	☐
☐	6. Does each analyte's ICAL include a minimum of 5 consecutive concentrations?	☐
☐	7. Was each standard concentration included in the ICAL?	☐
☐	8. If a point is dropped, is information noted in the ICAL explaining the reason?	☐
☐	9. Does this follow the Laboratory's point dropping policy? Are the injections dropped for that concentration for each analyte?	☐
☐	10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL?	☐
☐	11. For each analyte, are there no levels skipped?	☐
☐	12. For analytes calibrated using RF, is the RSD ≤15%? For Hydrogen ≥0.99?	☐
☐	13. For the ICV analysis, is the percent recovery for each analyte 85-115%?	☐
☐	14. Are all peak integrations including manual integrations (per *SOP for Manual Integration Policy*) acceptable? *If so, initial and date the appropriate pages.*	☐

COMMENTS:

_____

_____

_____

_____

Analyst _____ Secondary Reviewer _____

Date _____ Date _____

Attachment 3

Data Review Checklist

Fixed Gases per EPA Method 3C / ASTM D 1946 / ASTM D 1945
Data Review Checklist
(Note exceptions and include Sample Preparation and Analysis Observations / Case Narrative Summary Form as appropriate)

Analysis Date _____ Instrument ☐ GC19 ☐ GC ____

Client _____ QC Level _____

Project # _____ Due Date _____

Modification ☐ Yes ☐ No

Analyst **Reviewer**

Initial Calibration

☐ 1. Is the referenced ICAL the most recent ICAL performed? ..... NA

☐ 2. Has the referenced ICAL been peer reviewed and all associated documentation including the ICAL review checklist available for review? ..... NA

☐ 3. Were all associated requirements within the specified limits? ..... NA

Data

☐ 1. Is the sample data documentation present and correct? ..... ☐
- ☐ Sample raw data?
- ☐ All target analyte responses within calibration range?
- ☐ All peak integration acceptable?
- ☐ All manual integrations flagged and documented (before and after)? If so, initial and date.
- ☐ All analyte retention times within the generated RT window?
- ☐ All calculations correct?
- ☐ First quantitation report initialed and dated by analyst?

☐ 2. Do all sample duplicate injections (if analyzing without modification have a RSD ≤5%? ..... ☐

☐ 3. CCV have a percent difference of ≤10% (≤15% for hydrogen)? ..... ☐

☐ 4. Is the retention time (for CCV) for each analyte in the standard within 0.33min from the mean RT (of the corresponding analyte) from the ICAL? ..... ☐

☐ 5. Is the sum of the gases in the lab air within 90% and 110%? ..... ☐

☐ 6. Are the %R for the LCS within the acceptance criteria for each analyte? ..... ☐

☐ 7. Are the analytes in the MB < MRL? ..... ☐

☐ 8. Do all reported analytes fall within the generated retention time windows? If not, is the reason for reporting analyte in the sample documented? ..... ☐

☐ 9. Is the RPD (with modification) for the LD within the laboratory generated RPD limits? ..... ☐

☐ 10. **DOD: Are manual integrations notated in the case narrative**? ..... ☐

COMMENTS:

_____

_____

_____

☐ LIMS Run Approval ☐ LIMS Supervisor Approval

Analyst _____ Secondary Reviewer _____

Date _____ Date _____

Attachment 4

Method Reporting Limits and Control Limits

Target Analytes with Associated MRLs

Compound	Method Reporting Limit
Hydrogen	1000ppm
Oxygen	1000ppm
Nitrogen	1000ppm
Carbon monoxide	1000ppm
Methane	1000ppm
Carbon dioxide	1000ppm

Laboratory Generated Control Limits – ASTM D 1946-90 / Modified EPA 3C Single Injection

Analyte	LCS - LCL (%R)	LCS - UCL (%R)	LD (RPD)
Hydrogen – H_2	83	114	16
Oxygen – O_2	84	121	16
Nitrogen – N_2	88	122	21
Carbon monoxide - CO	87	118	16
Methane – CH_4	85	116	16
Carbon dioxide – CO_2	84	117	16

Note: New limits may be established prior to the revision of this document, refer to the most recent control limits.

Attachment 5

Calibration Curve Concentrations

Suggested Calibration Curve Concentrations (ppm unless noted as %)

ICAL	Hydrogen	Oxygen	Nitrogen	Carbon Monoxide	Methane	Carbon Dioxide
1	373.69	467.11	466.18	470.85	373.69	467.11
2	2000	2500	2495	2520	2000	2500
3	7473.77	9342.21	9342.21	9379.58	7511.14	9323.53
4	40000	50000	50000	50200	40200	49900
5	467731.47	584664.34	584664.34	587002.99	470070.13	583495
6	99.999%					
7		99.999%				
8			99.999%			
9					99.999%	
10						99.999%

ICAL	Amount of Standard Spiked onto Instrument
1	small loop injection of a 2500ppm/2000ppm standard [1,2]
2	standard loop injection of a 2500ppm/2000ppm standard [1,2]
3	small loop injection of a purchased 5%/4% standard (see section 10.3.1.1) [2]
4	standard loop injection of a purchased 5%/4% standard (see section 10.3.1.1) [2]
5	large loop injection of a purchased 5%/4% standard (see section 10.3.1.1) [2]
6 through 10	standard loop injection of neat gas compounds (see section 10.3.1.1)

[1]2500ppm/2000ppm standard is made by introducing 600ml of a purchased 5%/4% standard into a 6 liter summa canister and pressurized to +14.7psig (29.4psi) with helium.
[2]The loop injection volumes are calculated as described in section 12.14 and shown in Table 1.

Calibration Range	
Hydrogen	1000ppm – 99.999%
Oxygen	1000ppm – 99.999%
Nitrogen	1000ppm – 99.999%
Carbon Monoxide	1000ppm – 58.700%
Methane	1000ppm – 99.999%
Carbon Dioxide	1000ppm – 99.999%

ALS Standard Operating Procedure

Proprietary - Uncontrolled Copy

DOCUMENT TITLE:	DETERMINATION OF AIR-PHASE PETROLEUM HYDROCARBONS (APH) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCED METHOD:	MADEP APH
SOP ID:	VOA-MAPH
REV. NUMBER:	09.0
EFFECTIVE DATE:	03/21/2015

STANDARD OPERATING PROCEDURE

DETERMINATION OF AIR-PHASE PETROLEUM HYDROCARBONS (APH) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

MADEP APH

SOP ID:	VOA-MAPH	Rev. Number:	09.0	Effective Date:	03/21/2015

Approved By: _____ Date: 3/10/15
Technical Manager (Volatile GC/MS) - Chris Parnell

Approved By: _____ Date: 3/10/15
QA Manager - Chaney Humphrey

Approved By: _____ Date: 3/11/15
Laboratory Director - Kelly Horiuchi

Archival Date:	_____	Doc Control ID#:	Non-Controlled	Editor:	_____

TABLE OF CONTENTS

DETERMINATION OF AIR-PHASE PETROLEUM HYDROCARBONS (APH) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1) Scope and Applicability

1.1 This procedure is based on and incorporates the requirements detailed in the Method for the Determination of Air-Phase Petroleum Hydrocarbons (APH), Revision 1, December 2009, Massachusetts Department of Environmental Protection. It is designed to measure the gaseous-phase concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in air. Volatile aliphatic hydrocarbons are collectively quantitated within two carbon number ranges: C5 through C8 and C9 through C12. In addition, volatile aromatic hydrocarbons are collectively quantitated within the C9-C10 range. Also, this method may be used to measure the individual concentrations of target APH analytes 1,3-butadiene, methyl-tert-butyl ether (MtBE), benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, and naphthalene in air. An extended list of target analytes also may be reported since this method overlaps with EPA Method TO-15.

1.2 This method typically applies to whole air samples received in Summa stainless steel canisters, with subsequent analysis by gas chromatography/mass spectrometry (GC/MS). The method reporting limit (MRL) for this method for each of the collective aliphatic and aromatic fractional ranges is approximately 2.5 – 20ug/m3. The MRL for the target APH analytes is compound specific but is approximately 0.50ug/m3. Refer to the most recent method detection limit study and initial calibration for the corresponding method detection and reporting limits. The reported MRL may be adjusted higher; however, the capability of achieving lower MRLs for specific project requirements must be thoroughly demonstrated and documented. The number of samples that may be analyzed in a 24-hour period is about twenty.

2) Summary of Procedure

2.1 Samples are collected in pre-cleaned, evacuated Summa stainless steel canisters. An aliquot of an air sample is concentrated on a solid adsorbent trap to collect the analytes of interest. To remove co-collected water vapor, the concentrated sample then goes through a water removal (dry purge) step. After the sample is pre-concentrated on a trap, the trap is heated and the APHs are thermally desorbed onto a refocusing cold trap. The APHs are then thermally desorbed onto the head of a capillary column once the cold trap is heated. The oven temperature (programmed) increases and the APHs elute and are detected by the mass spectrometer. The GC/MS utilizes a linear quadrupole system, which allows for it to be operated by either continuously scanning a wide range of mass to charge ratios (SCAN mode) or by Select Ion Monitoring mode (SIM), which consists of monitoring a small number of ions from a specified compound list.

2.2 Target APH analytes are identified and quantitated using characteristic ions. Collective concentrations of C9-C10 aromatic hydrocarbons are quantitated using extracted ions. Collective concentrations of aliphatic hydrocarbons fractions are quantitated using a total ion chromatogram, subtracting out target APH analytes and C9-C10 aromatic hydrocarbons. The target analytes will be quantitated and reported using EPA method TO-15. Since the sample pre-concentration steps and analytical conditions are identical for TO-15 and the Massachusetts APH method, all sample results can be generated from the same analytical run.

3) Definitions

3.1 Cryogen A refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Liquid nitrogen (cryogen) is used for this purpose and it has a boiling point of -195.8°C.

3.2 Gauge Pressure Pressure measure with reference to the surrounding atmospheric pressure, usually expressed in units of psi. Zero gauge pressure is equal to atmospheric (barometric) pressure.

3.3 MS-SCAN Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

3.4 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.5 Stock Standard A purchased, multi-component gas-phase mixture having certified concentrations, used to prepare working calibration standards.

3.6 Working Calibration Standard A gas-phase mixture of all the target analytes at a known concentration prepared by diluting a gas-phase stock standard into a Summa canister. Used for calibrations. Standard canisters prepared from methanol stocks are not allowed.

3.7 Calibration or Standard Curve A calibration or standard curve is a graph which plots the concentration of a compound (or an analyte) versus the instrument response to the compound.

3.8 Initial Calibration Verification (ICV) Standard A gas-phase standard prepared in the laboratory containing known concentration(s) of analytes of interest. It is prepared from gas-phase stock standards which are from a different source than the standards used to prepare the working calibration standards. Standard canisters prepared from methanol stocks are not allowed.

3.9 Continuing Calibration Verification (CCV) Standard A working calibration standard which is analyzed at specific intervals in order to verify that the instrument continues to meet the calibration criteria.

3.10 Field Sample A sample collected and delivered to the laboratory for analysis.

3.11 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop "ticks" have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.12 Batch Quality Control (QC) Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD).

3.13 Internal Standard Calibration Compares the instrument responses from the target compound in the sample to the responses of specific standards (called internal standards), which are added to the sample or sample preparation prior to analysis. The ratio of the peak area (or height) of the target compound in the sample or sample preparation is compared to a similar ratio derived for each calibration standard.

3.14 May This action, activity, or procedural step is neither required nor prohibited.

3.15 Must This action, activity, or procedural step is required.

3.16 Shall This action, activity, or procedural step is required.

3.17 Should This action, activity, or procedural step is suggested, but not required.

3.18 Service Request A form generated, at the time of sample receipt, which details pertinent information such as client name, address, contact, client and laboratory sample identifications, sampling and receipt dates and times, requested analyses, sample type, canister pressures (initial and final), and the service request number (unique number for each submitted job) and serves as an inter-laboratory "custody" form which accompanies all samples throughout the laboratory.

3.19 Air-Phase Petroleum Hydrocarbons (APH) These are defined as collective fractions of hydrocarbons compounds eluting from isopentane to n-dodecane, excluding target APH analytes. APH is comprised of C5-C8 aliphatic hydrocarbons, C9-C12 aliphatic hydrocarbons, and C9-C10 aromatic hydrocarbons.

3.20 APH Component Standard A mixture of the aliphatic and aromatic compounds listed in Table 4. The compounds comprising the APH Component Standard are used to define and establish the retention time windows for the collective aliphatic and aromatic hydrocarbon ranges of interest, and determine average chromatographic response factors that can in turn be used to calculate the collective concentration of hydrocarbons within these ranges. The APH target analytes are in a separate stock standard cylinder (also used for EPA Method TO-15) and are prepared as separate working standards in Summa canisters.

3.21 Laboratory Control Sample A humidified canister fortified with a gaseous-phase mixture of the APH Component Standard obtained from a different stock solution than the APH working/calibration standards.

4) Health and Safety Warnings

4.1 Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, Safety Data Sheet (SDS) location, and the laboratory waste management plan for the safe disposal of chemicals and samples.

4.2 Pollution Prevention and Waste Management

All waste disposals shall be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal.* In addition, canisters must be cleaned in accordance with the requirements detailed in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters.*

4.3 This procedure may include CHEMICAL, OPERATIONAL and/or EQUIPMENT hazards. Employees must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1: Standard and Sample Preparation	**Hazards**	**Preventative Measures**
Compounds, mixtures of compounds, standards, surrogates, and samples.	Exposure to potential health hazards through absorption through skin. Inhalation hazards.	Reduce exposure through the use of gloves and fume hoods. Safety glasses must be worn when working in the prep lab. Care should be taken when handling standard material in a neat or highly concentrated form. Personal protective clothing (safety glasses, gloves, and lab coat) are required when handling standard material in neat form. Consult Safety Data Sheets (SDS) for compounds being handled in this procedure, and be familiar with proper safety precautions.
Job Task #2: Working with Liquid Nitrogen	**Hazards**	**Preventative Measures**
Turning valves and handling tubing and fittings that have been in contact with the cryogen.	Can cause serious tissue damage (frostbite) with only a few seconds of contact.	Wear neoprene or leather gloves. Valves on cryogen dewars should be opened slowly so leaky fitting can be identified.
Job Task #3: Working with Pressurized Gases	**Hazards**	**Preventative Measures**
Using and moving compressed gas cylinders.	Gas leak, fire, and explosion. Personal injury due to falling during transport.	All cylinders must be secured in an upright position to a wall or immovable counter with a chain or a cylinder clamp when not in use. Keep safety caps on when cylinders are not in use. A handcart must be used when transporting cylinders. The cylinder must be secured to the handcart with a chain or belt. The regulator should never remain on small "D" size cylinders following use. Full cylinders must be kept separate from empty cylinders. Flammable gases (i.e. pressurized hydrogen) must be clearly labeled. Flammables and oxidizers must be separated by a ½-hour fire wall or by at least twenty feet.
Job Task #4: Glass Syringes	**Hazards**	**Preventative Measures**
Glass syringe use	Skin lacerations and punctures.	The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

5) Cautions

5.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

The instrument maintenance log must be kept current. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The entry in the log must include:

(a) the date of maintenance
(b) who did the maintenance
(c) description of the maintenance
(d) proof that the maintenance activity was successful

A notation of a successful tune and continuing calibration or initial calibration and the file number that accompanies the data will serve as proof that the maintenance is complete and the instrument is in working order.

The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, changing the pump oil, cleaning the source, or ordering a part. In addition, a notation should be made in the logbook stating that no samples were analyzed during the days that the instrument was down and no active maintenance was being conducted (i.e., where no other notation was made in the logbook for those days).

5.2 Concentrating Trap Routine maintenance includes periodic solvent cleaning of the Silcosteel lines in the valve oven if contamination is suspected. Also, periodic replacement of the multi-sorbent or partial replacement of the trap if analyte specific deterioration is detected is required. After repacking the trap it should be baked for a minimum of two hours (until a clean blank is generated), whereas a partial repacking requires baking the trap for a minimum of 20 minutes (or until a clean blank is generated).

5.3 GC System Column performance is monitored by observing both peak shapes and column bleed. Over time, the column will exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced (see Section 9.4). Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.4 Mass Spectrometer The Mass Selective Detector (MSD) ion source requires periodic cleaning to maintain proper performance. Symptoms of a dirty ion source include difficulty keeping the MSD in tune and fluctuating internal standard areas. The vacuum system should be serviced every six months, including changing the pump oil and checking the molecular sieve in the backstreaming trap.

5.5 Instrument Tuning The instrument is tuned with guidance from the procedure described in the Agilent Operations Manual, when necessary. The tune shall meet the tune criteria described in this document.

6) Interferences

6.1 Summa Canisters Canisters should be stored in a contaminant free location and should be capped tightly during shipment to prevent leakage and minimize any compromise of the sample. The pressure/vacuum is checked prior to shipment and upon receipt from the field. Any problems with the sample from the field are noted on the service request form and the Project Manager contacted.

Also, canisters must be cleaned and certified to be free from target analytes before being shipped to the field for sample collection. The procedure is described in detail in the *Standard Operating Procedure for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* (refer to this procedure as well as Section 12.7.1 for the acceptance criteria.).

6.2 Analytical System The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with buna-N rubber components must be avoided.

6.3 Glassware Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware results in discrete artifacts and/or elevated baselines in the detector profiles should be minimized. All glassware associated with this method must be scrupulously cleaned to avoid possible contamination. The cleaning shall be performed in accordance with the procedure outlined in the *SOP for Glassware Cleaning.* The use of high purity water, reagents, and solvents helps to minimize these problems.

6.4 Organic Compounds Certain organic compounds not associated with the release of petroleum products, including chlorinated solvents, ketones and ethers will be detected by this method and quantified within an aliphatic or aromatic hydrocarbon range. *When noted by the analyst, the identification and/or quantitation of such compounds must be disclosed on the laboratory report.* Non-APH compounds may be subtracted out of the hydrocarbon ranges before reporting results. When requested by the data user the identification of such non-APH compounds must be disclosed on the laboratory report or case narrative.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP may perform analysis, and interpretation of the results. The analyst must also ensure that a second analyst that is familiar with this analysis reviews the results and all applicable QC.

7.2 The supervisor/manager must ensure that method proficiency is documented initially and whenever significant changes in the instrument type, personnel, matrix or test

method are made.

7.3 The department supervisor/manager or designee shall perform final review and sign-off on the data.

7.4 All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy.* The training plan (Attachment 1) shall be used to document the training certification of new analysts.

8) Sample Collection, Handling, and Preservation

8.1 Air samples are collected in the field and delivered to the laboratory and should be collected in a specially prepared, leak-free, stainless steel pressure vessel (with valve) of desired volume (e.g., 6L). It is also acceptable to use Bottle Vacs (Entech Instruments, Simi Valley, CA) which are specially treated amber glass bottles fitted with a fused silica-coated valve (typically one liter volume). The use of Tedlar bags is considered a modification and is discouraged due to the inherent chemical artifacts which can interfere with the analysis.

8.2 Time-integrated samples require the use of a properly calibrated flow controller (refer to the Standard Operating Procedure for Flow Controllers and Critical Orifices). The flow controller must be calibrated prior to sample collection. Upon receipt at the laboratory, a post sampling calibration check must be performed on the flow controller. The relative percent difference (RPD) between the initial and post sampling calibration readings must be calculated. As long as the RPD is ≤20%, the calibration is considered to still be valid and thus the sample collection interval is also assumed to be valid. If the RPD is >20%, consideration must be given to whether resampling is necessary to achieve data quality objectives. If the sample is analyzed, a notation must be provided on the data reporting sheet and case narrative disclosing the RPD value.

8.3 There are no special preservation requirements for canisters. Canisters should be stored on the appropriate shelves until they are to be analyzed. The required holding time for samples in canisters for this method is 30 days.

9) Equipment and Supplies

9.1 Gas Chromatograph (GC) An instrument capable of temperature programming, with a column oven that may be cooled to sub-ambient temperature at the start of the gas chromatographic run to result in the resolution of the VOCs.

9.2 Autosampler

Teledyne-Tekmar AutoCan Autosampler:	14-ACAN-074
Concentrating Trap (cryogenic trap, built-in):	14-6938-020
Cryofocusing Module w/split valve:	14-6520-A00
GAST Vacuum Pump:	DOA-P104-AA

9.3 Mass Spectrometer (MS) A MS capable of scanning from 33 to 350 amu every second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

9.3.1 Ionization Gauge Controller

Granville-Phillips 330 Ionization Gauge Controller:	330001/2/3
Hewlett Packard Ionization Gauge Controller:	59864B

9.4 Analytical Column

Restek Rxi-1ms Fused Silica Capillary Column
60m x 0.25mm ID
1.0 micron film thickness

NOTE: Based upon data obtained from the MADEP VPH Round Robin testing programs, the choice of chromatographic column may have a significant impact on the apportionment and quantitation of aliphatic and aromatic compounds within the fractional ranges specified in this method. Substitution of the required column is not allowed, unless it can be demonstrated that the selected column has equivalent chromatographic properties and elution order for the aliphatic and aromatic compounds and ranges of interest.

To demonstrate equivalency of column chromatography, a mid-range calibration standard must be analyzed on both the required column and the proposed substitute column, with all other run and system parameters held constant. The concentrations of C5-C8 and C9-C12 aliphatic hydrocarbons, C9-C10 aromatic hydrocarbon ranges and target analytes must be determined for each column. The relative percent difference between the concentrations of each hydrocarbon range and target analyte, excluding naphthalene, obtained from each column must be ≤25. The RPD for naphthalene must be ≤40. The elution order of APH Components on the proposed substitute column must be equivalent to the elution order on the required column.

9.5 Data Systems IBM-compatible PC with Windows 95/98/NT/XP and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library or equivalent.

9.6 Canister Pressurization Station Vacuum/Pressure Gauge [0 to –30 in Hg; 0-90 psig]

9.7 Canister Sampling Devices VICI Condyne Model 300 Flow Controller

9.8 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent

10) Standards and Reagents

10.1 Reagents

10.1.1 UHP Grade Helium (99.999%)(GC carrier gas and preconcentrator purge/sweep gas)
10.1.2 Cryogen - Liquid nitrogen (used to cool preconcentrator traps)
10.1.3 UHP/Zero Grade Air
10.1.4 ASTM Type II Water or equivalent
10.1.5 High purity grade methanol

10.2 Standards

10.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Mixture Prepare a standard solution of p-Bromofluorobenzene (BFB-used as both a tune check and surrogate compound), bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene, 1,2-dichloroethane-d4(surrogate), and toluene-d8(surrogate) at 500ug/m^3 each in humidified zero air or nitrogen. This mixture may be purchased from an approved vendor in a high-pressure cylinder at the working concentration and Summa canisters filled directly from it for use on the sample preconcentrator. Otherwise, prepare this standard

according to the procedure outlined in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.

10.2.1.1 An intermediate standard can be prepared from neat compounds in a glass static dilution bottle (SDB). After the volume of the SDB is determined, calculate the mass of each compound to be spiked to achieve a final concentration of 5.0ug/mL. Then use the density of each neat compound to calculate the microliter amount to be spiked into the SDB. The SDB is then heated for a minimum of one hour at ~60˚C to completely volatilize all components.

Concentration of the intermediate standard prepared in a SDB is 5.0µg/mL. The amount required to achieve this concentration is determined through the use of the following equation.

$$A = \frac{(C)(V)}{D} \qquad \text{(Equation 1)}$$

Where:

A Amount of each compound required to achieve the desired concentration of the standard in the SDB (µL)
C Desired concentration of SDB (µg/mL)
V Actual volume of the SDB (mL)
D Density of the compound in question (µg/µL)

Example:

Calculate the amount of neat bromochloromethane needed to achieve the final concentration of 5.0µg/mL of that compound in the SDB.

V = 2010mL
D = 1934.4µg/µL
C = 5.0µg/mL

$$A = \frac{\left(5.0\frac{\mu g}{mL}\right)2010mL}{1934.4\frac{\mu g}{\mu L}} = 5.2\mu L$$

Table 1 – Tune, IS and Surrogate Compound Densities

Density (µg/µL)	Compound
1934.4	Bromochloromethane
1170.1	1,4-Difluorobenzene
1157	Chlorobenzene-d5
1307	1,2-Dichloroethane-d4
943	Toluene-d8
1593	BFB

10.2.1.2 The Working standard is prepared in a Summa canister by spiking an aliquot of the stock SDB standard (8.2.1.1) using a heated gastight syringe. Connect a cleaned, evacuated Summa canister to a source of

pure diluent gas (humidified zero air) using a teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum. Spike the SDB stock and following removal of syringe a small flow of diluent gas to flush the spike into the can. Pressurize the can to positive 83.3 psig with humid zero air, and allow the contents to equilibrate for approximately 24 hours before using.

Concentration of the working standard prepared in a Summa canister is 500ng/L. The final pressure of the canister is 83.3psig; therefore, the pressurized volume is 40L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 2)

Where:

PV Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PF = $\frac{P_{atm} + P_f}{P_{atm} + P_i}$

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister @ 1atm

P_{atm} Atmospheric Pressure = 14.7psig

Example:

$\frac{14.7 + 83.3}{14.7 + 0}(6L)$ = 40L

In order to prepare the canister with a concentration of 500ng/L, it must be determined how much of the intermediate standard is required. This is achieved through the use of the following equation.

$$A = \frac{(F)(V)}{(C)\left(1000\frac{ng}{\mu g}\right)}$$ (Equation 3)

Where:

F Desired concentration of working standard (ng/L)

V Pressurized Volume of Canister (L)

C Concentration of prepared SDB (µg/mL)

A Amount of standard (mL) of the SDB required to obtain the desired working standard concentration

Example:

$$A = \frac{500\frac{ng}{L}(40L)}{\left(5.0\frac{\mu g}{mL}\right)\left(1000\frac{ng}{\mu g}\right)} = 4mL$$

10.2.1.3 Currently the working standard is purchased in a cylinder at a certified concentration of 500ng/L (prepared by Liquid Technology Corporation). The working standard is filled directly into a summa canister to a pressure of 70 to 80 psig.

10.2.2 APH Component Standard (Stock Standard) Stock standards are purchased from an approved vendor as a mixture in a balance gas of nitrogen in high-pressure inert cylinders, and are available from several vendors. Each standard cylinder must be accompanied by a certificate of analysis stating the certified concentrations of each component. These concentrations must be used as the starting point when calculating the nanogram on-column amounts for the initial calibration points. See Table 5.

10.2.3 APH Working Standards Prepare gaseous-phase APH Working Standards at a minimum of two concentration levels in 6.0L Summa canisters pressurized with humidified zero air to 14.7psig. The contents should be allowed to equilibrate for approximately 24 hours prior to use.

Step 1: Concentration of the working standards prepared in Summa canisters should be 200ng/L and 20ng/L. The final pressure of the canister is 14.7psig; therefore, the pressurized volume is 12L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 6)

Where:

PV Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PF = $\frac{P_{atm}+P_f}{P_{atm}+P_i}$

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister @ 1atm

EXAMPLE:

$$\frac{14.7+14.7}{14.7+0}(6L) = 12L$$

Step 2: Use the Entech dynamic diluter to prepare the working standards in Summa canisters. The stock standard is typically at a concentration of 1000ng/L, so a 200ng/L can will be a 5X dilution, and the 20ng/L can will be a 50X dilution. Instructions for using the diluter and calculating flows can be

found in the instruction manual and in the TO-15 SOP (VOA-TO15).

10.2.4 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) For the second-source standard, use the TO-15 second source working standard. This standard contains all of the target analytes and at least one calibration compound from each hydrocarbon range.

Note 2: Any of the desired standard concentrations may change as long as the equations and the appropriate densities remain the same. In addition, the SDB volumes will change with each specific SDB utilized (indicated by the etched volumes on the specific SDB being utilized). The final pressures of the canisters may also change as long as the actual pressurized volumes are properly calculated in accordance with the corresponding equations detailed in this document. Use this section to calculate the alternate concentrations, pressurized volumes of the Summa canisters, etc., as needed.

10.3 Storage and Expiration Dates

- Static Dilution Bottle (SDB) standards (internal standard/surrogate) must be stored in an oven at a temperature of 60°C to ensure analyte vaporization. Every time a standard is prepared from the static dilution bottle (SDB), the concentration changes. To increase the useful lifetime of an SDB standard, remove volumes of 25mL or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, a SDB intermediate standard is stable for approximately *two months* as long as new working standards made from this standard continue to meet acceptance criteria. These bottles must be in the oven at 60°C for a minimum of one hour prior to use in preparing working standards.
- Stock Standard cylinders – These standards have an expiration date on the certificate of analysis (typically one year). Expired cylinders with sufficient volume remaining are sent back to the original vendor for recertification.
- APH Working Standards (excluding the ICV/LCS) prepared in canisters may be stored at laboratory conditions for *two months* in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

11) Method Calibration

11.1 Initial Calibration The APH Component Standards are used to calibrate the GC/MS system. Two distinct calibration operations are necessary:

Target APH Analytes: Relative Response Factors (RRFs) are calculated for the 9 Target APH Analytes (Table 4) and internal standards, based upon a correlation between the mass of analyte and area counts for the relevant quantitation ions. This allows for the individual identification and quantitation of these specific compounds. IT IS NOT NECESSARY TO DEVELOP RESPONSE FACTORS FOR ANY OTHER INDIVIDUAL APH COMPONENT STANDARD. However, an extended list of target analytes may be reported if needed since all the APH target analytes are included in the calibration for EPA Method TO-15 which is performed using the same GC and data acquisition parameters as the hydrocarbon range calibration.

Collective Aliphatic/Aromatic ranges: Relative Response Factors are calculated for C_5-C_8 Aliphatic Hydrocarbons and C_9-C_{12} Aliphatic Hydrocarbons based upon a correlation

between the TOTAL mass of aliphatic APH Component Standards eluting within the range of interest and the total ion area count. A Relative Response Factor is calculated for C_9-C_{10} Aromatic Hydrocarbons based upon a correlation between the TOTAL mass of aromatic APH Component Standards eluting within this range and the total area count of extracted ions 120 and 134. Specified APH Component Standards are designated "marker" compounds to define the beginning and end of the hydrocarbon ranges.

Primary and secondary extracted ions for all APH Component Standards and recommended internal standards are provided in Table 4. The recommended internal standards and associated Target APH Analyte and Hydrocarbon Ranges are provided in Table 3.

Table 3

Internal Standards and Associated Target APH Analytes and Hydrocarbon Ranges

Bromochloromethane (IS #1)	1,4-Difluorobenzene (IS #2)	Chlorobenzene-d5 (IS #3)
1,3-Butadiene Methyl tert-Butyl Ether	Benzene Toluene C_5-C_8 Aliphatics	Ethylbenzene m&p-Xylenes o-Xylene Naphthalene C_9-C_{12} Aliphatics C_9-C_{10} Aromatics

Table 4
Primary (Quantitation) & Secondary Ions for APH Component/Internal Standards

APH Component Standard	CAS Number	Mol Wt. (g/mol)	Target APH Analyte	Quantitation Ion	Secondary Ion(s)
Bromochloromethane (IS1)	74-97-5			130	49, 130
1,3-Butadiene	106-99-	54.09	✓	54	53, 39
Isopentane (Range Marker)	78-78-4			43	42, 41, 57
Methyl-tert-butyl ether	1634-	88.15	✓	73	57, 45
n-Hexane	110-54-			57	41, 43, 56
Cyclohexane	110-82-			56	84, 41
1,4-Difluorobenzene (IS2)	540-36-			114	88
2,3-Dimethylpentane	565593			56	43, 57, 41
Benzene	71-43-2	78.11	✓	78	52, 51
n-Heptane	142-82-			43	71, 57,
Toluene	108-88-	92.14	✓	91	92, 65
Chlorobenzene-d5 (IS3)	3114-			82	117
n-Octane	111-65-			43	85, 57, 71
Ethylbenzene	100-41-	106.17	✓	91	106
2,3-Dimethylheptane	3074-			43	84,85
m-Xylene	108-38-	106.17	✓	91	106, 105
p-Xylene	106-42-	106.17	✓	91	106, 105
n-Nonane (Range Marker)	111-84-			43	57, 85
o-Xylene (Range Marker)	95-47-6	106.17	✓	91	106, 105
Isopropylbenzene	98-82-8			105	120
1-Methyl-3-ethylbenzene	620-14-			105	120
1,3,5-Trimethylbenzene	108-67-			105	120
n-Decane	124-18-			57	43, 71, 85
Butylcyclohexane	1678-			83	55, 82
p-Isopropyltoluene	99-87-6			119	105, 134
1,2,3-Trimethylbenzene	526-73-			105	120
n-Undecane	1120-			57	43, 71, 85
n-Dodecane (Range Marker)	112-40-			57	43, 71, 85
Naphthalene (Range Marker)	91-20-3	128.17	✓	128	127, 102

Table 5
Standard Concentrations of APH Component Standards for Target APH Analytes and Hydrocarbon Ranges for Initial Calibration

Range	APH Component Standards used to Establish Range Response Factor	Calib. Level	Working Std conc (ng/L)	Injection Volume	Approximate Concentration
C_5-C_8 Aliphatic Hydrocarbons	Isopentane n-Hexane Cyclohexane 2,3-Dimethylpentane n-Heptane n-Octane	1	20	25mL	0.50ng
		2	20	50mL	1.0ng
		3	20	250mL	5.0ng
		4	200	125mL	25g
		5	200	250mL	50ng
		6	200	500mL	100ng
C_9-C_{12} Aliphatic Hydrocarbons	2,3-Dimethylheptane n-Nonane n-Decane Butylcyclohexane n-Undecane n-Dodecane	1	20	25mL	0.50ng
		2	20	50mL	1.0ng
		3	20	250mL	5.0ng
		4	200	125mL	25ng
		5	200	250mL	50ng
		6	200	500mL	100ng
C_9-C_{10} Aromatic Hydrocarbons	Isopropylbenzene 1-Methyl-3-ethylbenzene 1,3,5-Trimethylbenzene 1,2,3-Trimethylbenzene p-Isopropyltoluene	1	20	25mL	0.50ng
		2	20	50mL	1.0ng
		3	20	250mL	5.0ng
		4	200	125mL	25ng
		5	200	250mL	50ng
		6	200	500mL	100ng
Target APH Analytes	1,3-Butadiene Methyl tert-Butyl Ether Benzene Ethylbenzene m,p-Xylenes[b] o-Xylene Naphthalene	1	20	25mL	0.50ng
		2	20	50mL	1.0ng
		3	20	250mL	5.0ng
		4	200	125mL	25ng
		5	200	250mL	50ng
		6	200	500mL	100ng

[a] The actual concentrations shall depend on the certified analyte concentration from the applicable manufacturer's certificate of analysis.
[b] Xylene concentration is doubled.

11.1.1 Calibration Points Analyze a minimum of five levels of the calibration standard (analyze low to high) that span the monitoring range of interest of the samples. The range is typically 0.50ng to 100ng on column (m,p-Xylene is doubled). The dynamic range is dependent on the sensitivity of a particular instrument as well as the required reporting limit for a given project and may be adjusted accordingly. Refer to Table 5 for the approximate concentrations of the compounds of interest in the initial calibration. These concentrations may

change with the purchase and/or preparation of new standards; therefore, they should be verified.

The initial calibration is performed to determine instrument sensitivity and the linearity of the GC/MS response for the target compounds. One of the calibration points from the initial calibration curve must be at the same concentration as the continuing calibration verification standard. Also, one of the standards must be at or below the method reporting limit for the compounds of interest or the MRL must be adjusted accordingly.

11.1.2 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument or if the continuing calibration verification acceptance criteria have not been met as specified in Section 16.6.4.

11.1.3 Analytical Window If time remains in the 24-hour tune window after meeting the acceptance criteria for the initial calibration, samples may be analyzed according to the procedure described in this document. If time does not remain in the analytical window, a new sequence shall commence with the analysis of the instrument performance check compound (BFB) and the continuing calibration verification standard.

11.1.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 12.3. Use the standards prepared in accordance with Section 10 of this SOP. Attach the calibration standard and internal standard canisters to the designated inlets on the preconcentrator and open the canister valves. Analyzing different volume aliquots of the calibration standards produces differing concentrations. Internal standards must be added at the same volume for every standard, sample and QC sample.

Analyte responses (target ion areas) are tabulated and recorded using the Enviroquant program. Quantitation ions for the target compounds are shown in Table 4 and the primary ion should be used unless interferences are present, in which case the secondary ion may be used.

11.1.5 Initial Calibration Requirements

Initial calibration requirements are as follows:

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. Highest concentration, together with the lowest concentration, defines the calibration range.
3. Lowest concentration must be at or below the method reporting limit.
4. A blank should be analyzed prior to beginning the analysis of the calibration standards.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from low to high concentration.
8. All ICAL analyses must be completed within the 24-hour tune window.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at or below the MRL and may not be

dropped unless the MRL is changed to the concentration of the remaining lowest standard.

- Points at the high end may be dropped, but doing so lowers the calibration range.
- Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).
- Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.
- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 24 hours).
- Once the ICAL has been used to calculate and report sample results, it is not to be changed.

11.1.6 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not met and at least annually. The following procedure must be followed when updating an initial calibration method.

1. Open the most recent method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID must be the date files were analyzed.
3. Quantitate midpoint standard and check retention times and integrations. Update retention times if necessary using QEdit or Easy ID (Tools → Easy ID). Requant if any changes are made and verify all peaks are identified correctly. Print.
   a. While midpoint standard is loaded update reference spectra (Continuing Calibration → Update Reference Spectra).
   b. With midpoint standard loaded update qualifier ion ratios and retention times (Initial Calibration → Update Levels → Select Update Level and then select Retention Times (Replace) and Replace Qualifier Ion Relative Responses).
   c. If necessary adjust integration parameters prior to processing remaining ICAL points.
4. Quantitate remaining ICAL standards. Review each peak for retention time, integration, and print. Review low level standards for acceptable signal to noise ratios and high level standards for saturation.
5. All responses must be cleared from ICAL before updating (Initial Calibration → Clear All Calibration Responses).
6. Update responses for each standard level (Initial Calibration → Update Levels) or (Initial Calibration → Quick Levels Update). If Quick Levels Update is used do not requant datafiles.

7. Save method.
8. Check Response Factor Report and evaluate whether any points should be dropped following the criteria outlined in this SOP.
9. Save method if any changes are made.
10. Verify calibration files listed on Response Factor Report are correct.
11. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report.

11.1.7 Initial Calibration Review Analyst's calculation and assessment along with a peer review of all ICAL data and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples. In the case where samples are placed on the autosampler and allowed to run overnight, the sample results may only be reported if the ICAL is reviewed and found to be acceptable. The ICAL checklist in Attachment 2 must be used to document the review and approval process.

Analyte concentrations, which are not "real", not to be reported, or otherwise marked off the initial calibration, should be followed by a short explanation regarding the reason for the omission.

11.1.8 Initial Calibration File An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- BFB tune analysis report
- Blank analysis quantitation report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and %recovery report.

11.2 Initial Calibration Verification Standard Verify the initial calibration by analyzing an initial calibration verification standard (ICV). This standard shall be obtained or prepared from materials acquired from a different manufacturer or lot from that of the initial calibration and prepared according to Section 10.2.4. At a minimum, it must contain 1,3-butadiene, benzene, toluene, ethylbenzene, m-&p-xylene, o-xylene, and naphthalene, and at least one compound from each hydrocarbon range. Methyl tert-butyl ether may be included but may have wider recovery acceptance limits.

Inject 25ng or less (refer to the appropriate manufacturer's certificate of analysis for the actual secondary source standard concentrations) of the ICV standard depending on the dynamic range of a given instrument.

12) Sample Preparation/Analysis

12.1 Sample Preparation and Leak Check The initial pressure/vacuum is checked and the canister pressurized as needed upon receipt by the laboratory. Samples collected in canisters shall be pressurized with humidified zero grade air or Nitrogen. However, if the samples are to be analyzed in accordance with EPA Method 3C then the samples must be pressurized with UHP Helium. The client must be made aware of this in advance and given the option of either submitting two canisters for analysis or receiving a report with qualified results.

Canister Pressurization Samples must be pressurized (to approximately 3.5psig) prior to analysis with humidified zero air (refer to exception stated above). This may be

accomplished by connecting the sample canister to a source of pure diluent gas (zero air) using a teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum and injecting 100uL of water into the can through the septum and allowed to vaporize for approximately 10 minutes. Alternatively, pressurize at a fill station by bubbling the diluent gas through a zero air bubbler. Both of these procedures shall utilize ASTM Type II water or equivalent. Additional information may be found in the *Standard Operating Procedure for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters*. Initial and final pressures shall be recorded and the dilution factor created by filling the sample canister is calculated using Equation 25 in Section 15.3.4.

Leak Check Connect the canister(s) to the autosampler. Place a ¼" stainless steel nut and ferrule on the inlet line facing the canister. Push the inlet line into the orifice of the canister and hold in place while tightening the fitting finger tight. Turn the stainless steel nut ¼ turn more with a wrench. The canister valves should be closed at this point. For Bottle Vacs, connect the female Micro-QT fitting to the autosampler. A leak check must be performed before connecting the sample bottles since the valve is open as soon as the bottle is connected.

Leak Checks – Leak check all canister inlet connections. Analysis may not begin until the leak check has passed for each canister being tested. If a leak is detected, it should be confirmed by placing on a different location. In addition, the valve threads should be inspected for defects which may prevent a good seal with the AutoCAN. Once a canister has "failed" the leak check it must be tagged, an NCAR initiated, and the PM notified. Regardless of what the client or PM specifies as the fate of the sample, the canister must be put on maintenance hold to complete a full 24-hour leak check. A yellow sheet is to be completed in addition to, but not in lieu of an NCAR. This is a fixed QA procedure with no allowance for deviation.

12.2 Analytical Sequence

12.2.1 Analytical Sequence For this internal standard calibration method analysis, a CCV standard is to be analyzed every 24 hours. That is, the last analysis in the sequence must be started within 24 hours from the time of the initiation of the sequence. The initiation is considered to be the injection of the BFB tune standard.

The analytical sequence must be completed for the analysis of ≤20 field samples. A method blank (MB) shall be run to monitor for laboratory introduced contamination. There must be at a minimum a laboratory duplicate (LD) analyzed in each batch to access batch precision. A laboratory control sample (LCS) shall be analyzed at a rate of at least one per batch of twenty or fewer samples. The concentration of the LCS (ICV standard) should be at the lower end of the calibration curve as an indication that the system allows for good recovery at those concentrations. The following is the analytical sequence guideline for this method.

Analytical Sequence Guideline

With Calibration	Tune Check[1]
	Calibration Standards (5 Standards Minimum)
	ICV Standard[2] (Acts as the ICV and LCS)
	QC Canister Checks[6]
	MB[7]
	Sample(s)
	Laboratory Duplicate[4]

With Continuing	Calibration Tune Check[1]
	CCV Standard[5]
	QC Canister Checks[6]
	MB[7]
	LCS[3]
	Sample(s)
	Laboratory Duplicate[4]

[1] The introduction of the tune check standard is the start of the 24 hour analysis window. The instrument performance check solution must be analyzed initially and once per 24 hour time period of operation.
[2] In this scenario, the ICV may also be evaluated as the LCS.
[3] An LCS shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard.
[4] A laboratory duplicate must be analyzed at a rate of 1 per 20 or fewer samples. The duplicate must be reported even if it is a batch duplicate.
[5] A CCV must be analyzed at the beginning of every analytical sequence.
[6] Any number of QC check canisters may be analyzed in the sequence to determine a canister cleaning batch or batches acceptability.
[7] Any of the QC Check Canisters may serve as the method blank as long as the minimum requirements detailed in this document are met. A method blank shall be analyzed at a rate of 1 in 20 or fewer samples.

12.3 Conditions

12.3.1 Sample Collection Conditions The suggested settings and system parameters are as follows:

Adsorbent Trap

Set Point:	40˚
Sample Volume:	25ml to 1,000ml
Dry Purge:	300mL
Sampling Rate:	100ml/min or 40ml/min
Desorb Temp.:	210˚C
Desorb Flow Rate:	8-10mL/min He
Desorb Time:	3.0 minutes

Refocusing Trap

Temperature:	-175˚C
Injection Temp.:	150˚C
Injection Time:	1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature:	10°C above desorb temperature
Initial Bakeout:	2 hours or until clean blank is obtained
After each run:	10 minutes

12.3.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

Item	Condition
Carrier Gas	Helium
Flow Rate	1.0-1.6mL/minute
Temperature Program	Initial Temperature: 10˚C Initial Hold Temperature: 1 minute Ramp Rate: 5˚C/min to 50˚C 2nd Ramp: 10˚C/min to 100˚C 3rd Ramp: 20˚C/min to 240˚C for 4 min hold
Detector B (MSD *Interface)*:	260˚C
Electron Energy	70 Volts (nominal)
Mass Range	33 to 280 amu (SCAN mode)
Scan Time	To give at least 10 scans per peak, not to exceed 1 second per scan.

12.4 Retention Time Windows The laboratory should calculate retention time windows initially and whenever a new GC column is installed. The laboratory must retain these data.

Before establishing retention time windows, ensure that the GC/MS system is operating within optimum conditions. Analyze an APH Calibration Standard on three separate occasions throughout the course of a 72-hr period. Serial analyses over less than a 72-hr period may result in retention time windows that are too restrictive.

Calculate the standard deviation of the three absolute retention times for each Target APH Analyte, range "marker" compound, internal standard, and MS tuning standard.

The retention time window is defined as plus or minus three times the standard deviation of the absolute retention times for each analyte of interest. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

In those cases where the standard deviation for a particular standard approaches zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop an operational retention time window.

Table 2
APH Range "Marker" Compounds and Range Retention Time Windows

Hydrocarbon Range	Beginning Marker	Ending Marker
C_5-C_8 Aliphatic	0.1 min. before isopentane	0.01 min. before n-nonane
C_9-C_{12} Aliphatic Hydrocarbons	0.01 min. before n-nonane	0.1 min. after naphthalene*
C_9-C_{10} Aromatic Hydrocarbons	0.1 min. after o-xylene	0.1 min before naphthalene

*The method specifies using n-dodecane as this marker, but in practice naphthalene elutes after n-dodecane so the laboratory must use naphthalene as the marker.

The relative retention time (RRT) and RRT window for each Target APH Analyte, internal

standard, and hydrocarbon range "marker" compound must be verified on a daily basis. The RRT for each analyte of interest shall be established as the midpoint of the window. The retention time window equals the midpoint ± three times the standard deviation (Equation 9).

12.5 Instrument Performance Check Since the BFB tuning compound is included in the internal standard canister and a autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

The following is the procedure to follow when performing the instrument performance check.

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.

All subsequent standards, samples and QC samples associated with a BFB analysis must use identical instrument conditions. Refer to Section 16.6.1 (Table 7) for the acceptance criteria and required corrective action.

12.6 Continuing Calibration Verification Standard Verify the calibration each working day, where necessary (e.g., an ICAL was not analyzed or the 24-hour tune window has closed) by analyzing a continuing calibration verification (CCV) standard from the initial calibration standard canister. The concentration of the calibration verification should be varied within the established calibration range.

12.7 Canister Quality Control Check and Method Blank A Quality Control (QC) check canister may also serve as a method blank (see note 1 below) as long as the analyte concentration requirements stated in the canister quality control check section (Section 12.7.1) and the other requirements (refer to Section 16.6.7 for internal standard requirements) are met. If a QC canister fails with respect to the analyte concentration criterion, it may still be used as a method blank as long as the method blank criteria stated in 12.7.2 are met. If a QC canister still fails, another QC canister or a new canister must be prepared and analyzed (per Section 12.7.2) in order to verify that no system contamination exists.

Note 1: The use of a QC canister as a method blank is considered acceptable since a canister that has been sent into the field, returned and cleaned more closely resembles the manner in which client samples are handled.

12.7.1 Canister Quality Control Check The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *Standard Operating Procedure for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included.

The canister to be checked, shall be pressurized with humidified zero grade air prior to analysis. Analyze an aliquot of one liter along with the same volume of internal standard as standards and samples. The unique laboratory barcode given to a canister shall be the information included in the sample analysis

identification, which is for tracking purposes. A canister is considered "clean" if the analysis shows <0.2ppbv of any target analyte or hydrocarbon range (refer to Note 1).

12.7.2 Method Blank In order for a method blank to be considered acceptable all target analytes must be less than the method reporting limit and fulfill the additional requirement in Section 16.6.5. If the QC canister(s) fail the corresponding criteria then the following must be performed.

- Prepare a canister that has not left the building by pressurizing with humidified zero air.
- Analyze an aliquot of the blank (1 liter) with internal standard
- Be consistent with the volume of internal standards introduced for each analysis.

Additionally, analyze a method blank whenever a high concentration sample is encountered and carryover is suspected.

The analyst should cross out those concentrations that are not real and initial and date the quantitation report for those QC Check canisters and method blanks that meet the acceptance criteria included in this section.

12.8 Laboratory Control Sample The laboratory control sample is an injection of the initial calibration verification standard. Inject the LCS (ICV) at concentrations at or below the midpoint of the calibration curve. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

12.9 Sample Analysis Prior to analysis, all sample containers should be at temperature equilibrium with the laboratory.

- Attach sample canisters Tekmar AUTOCan using a 9/16" wrench. Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments).
- Before opening the valve, check for leaking fittings by running the leak check program in the Teklink software. Quick connect fittings must be leak checked before connecting the sample container.
- If system is leak tight, open the canister valves and start the automated preconcentration procedure. Make sure the Chemstation data acquisition software has been readied.
- Maintain the trap at an elevated temperature until the beginning of the next analysis.
- Introduce the same volume of internal standards as used for the standards and QC samples.

Note 1: The secondary ion quantitation is only allowed if there is sample matrix interference with the primary ion. If the secondary ion quantitation is performed, document the reasons in the instrument run logbook and/or on the quantitation report (initial and date any notation).

Note 2: Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fitting higher than 80ºC.

12.9.1 Qualitative Identifications The Target APH Analytes must be identified by an analyst competent in the interpretation of chromatograms and mass spectra.

Two criteria must be satisfied to verify the identification: (1) elution of the component in the sample at the same GC relative retention time (RRT) as the component in the standard, and (2) agreement of the sample component and standard component mass spectra.

If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned using extracted ion current profiles for the ion unique to the component of interest.

For comparison of the standard and sample component mass spectra, mass spectra of standards obtained on the GC/MS under the same instrument conditions are required. Once obtained, these standard spectra may be used for identification and reference purposes. The requirements for qualitative verification by comparison of mass spectra are as follows:

All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

The relative intensities of ions specified must agree within ±20% between the standard and sample spectra.

Ions greater than 10% in the sample spectrum must be considered and accounted for by the analyst making the comparison.

The primary and secondary ions for all APH Component Standards are provided in Table 4.

12.9.2 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot should be analyzed. The smallest volume used shall not be less than that used for the initial calibration (see Table 5). The dynamic range of volume aliquots for the automatic cryogenic concentrator is 15ml to 1L. If a volume smaller than 15ml is to be analyzed, a dilution should be made in a Tedlar bag, or the sample directly injected using a gastight syringe. Guidance in performing dilutions and exceptions to this requirement are given below.

- Use results of the original analysis to determine the approximate dilution factor required and get the largest analyte peak within the initial calibration range.
- The dilution factor chosen should keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument.
- All dilution factors (Equation 25) must be documented and included in the final report.

Note: Refer to Section 18.7.3 for requirements on reporting results outside of the initial calibration range.

12.10 Manual Integration The integration for each peak shall be checked to ensure that it has been integrated properly. Assuming an incorrect automatic integration the analyst shall conduct the manual integration in accordance with the *SOP for Manual Integration Policy* including all documentation and reviews associated with the process. The review shall include the analyst and reviewer initialing and dating the manual integration as an indication of acceptability and approval.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Data System Setup

For the Tekmar AutoCan, fill in the sequence log of the Teklink program with the appropriate information. Refer to the Section 12.3.1 for the operating parameters.

For the HP Chemstation, load the appropriate acquisition method for the GC/MS in the top window of the Chemstation program. Suggested GC/MS operating parameters are given in Section 12.3.2.

14.2 Storing Electronic Data The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. Therefore, files will be named with an eight-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files are saved in a unique sub-directory on the server.

14.3 Sufficient raw data records must be retained of the analysis, instrument calibrations and method detection limit studies including: analysis/calibration date and time, test method, instrument, sample identification, analyte identification, analyst's initials, concentrations and responses, as well as standards used for the analysis and calibrations, all manual calculations including sample dilutions and manual integrations to permit reconstruction of analyses. Information entered and reported on the quantitation report and instrument run log must be complete and accurate. Retain all daily QC per sequence on file for future reference including tune checks, opening standards, method blanks, laboratory control samples, laboratory duplicates, and initial calibrations and initial calibration verifications. Additionally, all passing QC Canister checks must also be retained on file.

Note: All data records must explicitly connect data to the initial instrument calibration. This includes all samples, continuing calibrations and QC samples.

14.4 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings and balance gas if pressurized with helium), standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 Initial Calibration Calculations

15.1.1 Target APH Analytes Quantitation of the target analytes is done using the same data analysis method used for EPA TO-15 since all the APH target analytes are part of the laboratory's TO-15 analyte list. Tabulate the area response of the characteristic ions against the mass of each Target APH Analyte and internal standard and calculate relative response factors (RRFs) for each compound

using Equation 12. Perform this calculation for each Target APH Analyte.

Equation 12: Relative Response Factor for Target APH Analytes

$$RRF = [(A_{EC})*(C_I)] / [(A_{EI})*(C_c)]$$

where:

RRF = relative response factor
A_{EC} = area count of the extracted ion for the analyte of interest
C_I = mass of internal standard (ng)
A_{EI} = area count of the extracted ion for the associated internal standard
C_C = mass of analyte of interest (ng)

15.1.2 Hydrocarbon Ranges Calculate a response factor for the C_5-C_8 Aliphatic Hydrocarbon range using the following steps.

Using total ion integration, sum the individual peak areas of the six (6) APH Component Standards that are used to establish an average range response factor for C_5-C_8 Aliphatic Hydrocarbons, as designated in Table 5. Do not include the peak areas of internal/tuning standards.

Using the total area generated, calculate the Range RRF using Equation 13.

Equation 13: Relative Response Factor for C_5-C_8 Aliphatic Hydrocarbons

$$\text{Range } RRF = [(A_T)*(C_I)] / [(A_{EI2})*(C_T)]$$

Where:

Range RRF = relative response factor for the hydrocarbon range
A_T = total ion area count of the six aliphatic APH Component Standards which elute within this range (see Table 5)
C_I = mass of internal standard #2, ng (1,4-Difluorobenzene)
A_{EI2} = area count of the extracted ion for internal standard #2
C_T = summation of the masses of the six aliphatic APH Component Standards (ng) which elute within this range (see Table 5)

15.1.2.1 Calculate a response factor for the C_9-C_{12} Aliphatic Hydrocarbon range using the following steps.

Using total ion integration, sum the individual peak areas of the six (6) APH Component Standards that are used to establish an average range response factor for C_9-C_{12} Aliphatic Hydrocarbons, as designated in Table 5. Do not include the peak areas of internal/tuning standards.

Using the total area generated, calculate the Range RRF using Equation 14.

Equation 14: Relative Response Factor for C_9-C_{12} Aliphatic Hydrocarbons

$$\text{Range } RRF = [(A_T)*(C_I)] / [(A_{EI3})*(C_T)]$$

where:

Range RRF = relative response factor for the hydrocarbon range
A_T = total ion area count of the six aliphatic APH Component Standards which elute within this range (see Table 5)
C_I = mass of internal standard #3, ng (Chlorobenzene d5)
A_{EI3} = area count of the extracted ion for internal standard #3
C_T = summation of the masses of the six aliphatic APH Component Standards (ng) which elute within this range.

15.1.2.2 Calculate a response factor for the C_9-C_{10} Aromatic Hydrocarbon range using the following steps.

Using extracted ion 120, sum the individual peak areas of the five (5) APH Component Standards that are used to establish an average range response factor for C_9-C_{10} Aromatic Hydrocarbons (only four of the compounds will contribute area from m/z 120), as designated in Table 5. Do not include the peak areas of internal/tuning standards.

Using extracted ion 134, sum the peak areas of the five (5) APH Component Standards that are used to establish an average range response factor for C_9-C_{10} Aromatic Hydrocarbons (only one compound will contribute area from m/z 134), as designated in Table 5. Do not include the peak areas of internal/tuning standards.

Sum the area counts from each extracted ion.

Using the area count generated, calculate the RRF using Equation 15.

Equation 15: Relative Response Factor for C_9-C_{10} Aromatic Hydrocarbons

$$\text{Range } RRF = [(A_T)*(C_I)] / [(A_{EI3})*(C_T)]$$

where:

Range RRF = relative response factor for the hydrocarbon range
A_T = summation of area counts using extracted ions 120 and 134
C_I = mass of internal standard #3, ng (Chlorobenzene d5)
A_{EI3} = area count of the extracted ion for internal standard #3
C_T = summation of the masses of the five aromatic APH Component Standards (ng) which elute within this range (see Table 5)

Calculate the average response factor for each of the Target APH Analytes and each hydrocarbon range.

Calculate the percent relative standard deviation (%RSD) of the response factors over the working range of the curve for each of the Target APH Analytes and each hydrocarbon range using Equation 16.

Equation 16: Percent Relative Standard Deviation

This equation is also used for initial demonstration of capabilities, method detection limits studies, and method reporting limit verifications.

$$\%RSD = [(SD_{n-1}) / (AVG_X)]*100]$$

where:

%RSD = percent relative standard deviation
SD_{n-1} = standard deviation (n-1 degrees of freedom)
AVG_x = average response factor from the initial calibration curve

15.2 Sample Calculations

15.2.1 Individual Target APH Analytes The average response factor from the initial calibration is used to calculate the amount of analyte detected in the sample. Equation 17 is used to calculate the mass of sample analyte in ng. Equation 18 is used to convert ng to µg/m³. Equation 19 is used to convert of µg/m³ to ppbV.

Equation 17: Calculation of Analysis Results in ng

$$ng = [(A_x)*(C_{IS})] / [(A_{IS})*(RRF_{avg})]$$

where:

A_x = area of quantitation ion for the Target APH Analyte (see Table 4)
C_{IS} = mass of the internal standard
A_{IS} = area of quantitation ion for the associated internal std (see Table 4)
RRF_{avg} = average response factor for the specific compound to be measured**

Equation 18: Conversion of ng to µg/m³

$$\mu g / m3 = (ng / VA) * DF$$

where:

V_A = volume of sample analyzed (liters)
DF = dilution factor (Equation 25); if no dilution was made, the dilution factor =1

Equation 19: Conversion of µg/m³ to ppbV

$$ppbV = (\mu g / m3) * 24.46 / MW$$

where:

MW = molecular weight of the compound of interest, g/mol (see Table 4 for a list of the molecular weights of the Target APH Analytes)

15.2.2 Hydrocarbon Ranges The average range response factor from the initial calibration is used to calculate the mass (ng) of range hydrocarbons in samples. Collective peak area integration for the hydrocarbon ranges must be from baseline to baseline (i.e., must include the unresolved complex mixture).

15.2.3 The contribution of compounds not meeting the definition of aromatic or aliphatic hydrocarbons may be omitted from the collective hydrocarbon range calculations at the discretion of the laboratory and the data user. Only peaks with a peak height greater than half of the nearest internal standard need to be evaluated for exclusion. The guidance for making this decision includes the following:

- If the non-APH compound co-elutes with an aliphatic petroleum hydrocarbon, the area may not be subtracted from the aliphatic range.

- In complex sample matrices (i.e. many co-eluting peaks, complex petroleum patterns), this type of data adjustment may not be possible.
- Spectral identification of the excluded peak must be evaluated by a qualified mass spectrometrist. The analyst should consider the quality of the spectral library match, presence and relative intensity of major ions, and potential interferences in making a professional judgment on exclusion.

C_5-C_8 Aliphatic Hydrocarbons

- Using total ion integration, sum all peaks in the appropriate retention time window as specified in Sections 12.4 and Table 2.
- From this sum, subtract the total ion area counts of all internal standards and surrogates which elute in this range (all three of the recommended internal standards and two of the surrogates elute in this range). Also subtract the total ion area counts of all non-APH compounds that are not to be included in the final result.
- Calculate a preliminary mass amount in ng using Equation 20.

  Equation 20: Calculation of Preliminary Sample Analysis Results (ng)

  $$ng = [(A_x)*(C_{IS})]/[(A_{IS})*(RRF_{avg})]$$

  where:

  A_x = total ion area count of all peaks eluting within C5-C8 Aliphatic Hydrocarbon range window
  C_{IS} = mass of the internal standard, ng
  A_{IS} = area of quantitation ion for internal standard #2 (1,4-Difluorobenzene)
  RRF_{avg} = average range response factor for the C5-C8 Aliphatic Hydrocarbon range

- From the preliminary amount (ng), calculate an adjusted mass amount of range hydrocarbons by subtracting the masses of Target APH Analytes which elute in this range (MtBE, benzene, toluene, ethylbenzene, m-xylene, p-xylene, and o-xylene,).
- Convert the adjusted ng value to µg/m³ using Equation 21.

  Equation 21: Conversion of ng to µg/m³

  $$\mu g/m^3 = (C_{ng}/V_A)*DF$$

  where:

  C_{ng} = adjusted total mass of range hydrocarbons in ng
  V_A = volume of sample analyzed (liters)
  DF = dilution factor (Equation 25); if no dilution was made, the dilution factor = 1.

C_9-C_{10} Aromatic Hydrocarbons

- Using extracted ion 120, sum all peaks in the appropriate retention time window as specified in Section 12.4 and Table 2.
- Using extracted ion 134, sum all peaks in the appropriate retention time window as determined in Section 12.4 and Table 2.
- Sum the areas of ions 120 and 134.

- Subtract the extracted ion area (mass 120 and 134) of any non-APH compounds that are not to be included in the final result.
- Calculate an amount in ng using Equation 20, using the summed areas of ions 120 and 134.
- Convert the ng value to µg/m³ using Equation 21.

C_9-C_{12} Aliphatic Hydrocarbons

- Using total ion integration, sum all peaks in the appropriate retention time window as specified in Section 12.4 and Table 2.
- From this sum, subtract the total ion area counts of the 4-bromofluorobenzene (Surrogate #3) peak.
- Subtract the total ion area counts of all non-APH compounds that are not to be included in the final result.
- Calculate a preliminary mass amount in ng using Equation 20.
- From the preliminary amount (ng), calculate an adjusted mass amount of range hydrocarbons by subtracting the masses of Target APH Analytes which elute in this range (naphthalene), and by subtracting out the mass amount of C_9-C_{10} Aromatic Hydrocarbons.
- Convert the ng value to µg/m³ using Equation 21.

15.3 Additional Calculations

15.3.1 Relative Percent Difference This equation is used for laboratory duplicates and post calibration check of flow controllers when they are received back by the laboratory following sampling.

Equation 22: Relative Percent Difference

$$\frac{x_1 - x_2}{\bar{x}} \ (100)$$

where:

x_1 First measurement value
x_2 Second measurement value
$\bar{x}$ Average of the two values

15.3.2 Percent Difference This equation is used for the continuing calibration verification standards.

Equation 23: Percent Difference

$$\%D = [(RF_C) - (RF_I)] / [(RF_I)] * 100$$

where:

%D = percent difference
RF_C = response factor from the continuing calibration verification standard
RF_I = average response factor from the initial calibration curve

15.3.3 Percent Recovery This equation is used for the initial calibration verification standard, laboratory control sample, initial demonstration of capability, method detection limit study, and method reporting limit verifications.

Calculate the percent recovery (%R) of the Target APH Analyte or hydrocarbon range using Equation 24.

Equation 24: Percent Recovery

$$\%R = [(C_{found}) / (C_{true})]*100$$

%R = percent recovery
C_{found} = mass of the analyte or hydrocarbon range detected in the standard (ng)
C_{true} = true mass of the analyte or hydrocarbon range in the standard (ng)

15.3.4 Dilution Factors

Equation 25: Dilution Factor for Pressurization of Subatmospheric Samples:

$$PDF = \frac{P_{atm} + P_f}{P_{atm} + P_i}$$

where:

P_{atm} is the ambient atmospheric pressure, 14.7 psi at sea level.
P_f is the final sample canister pressure, in psig.
P_i is the initial sample canister pressure, in psig. This will most often be a negative value (sub-ambient initial pressure.)

15.3.5 Relative Retention Time

Equation 26: Relative Retention Time (RRT)

$$RRT = \frac{RT_C}{RT_{is}}$$

where:
RT_C Retention time of the target compound, seconds.
RT_{is} Retention time of the internal standard, seconds.

15.4 Data Review The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated by analytical sequence following the data review checklist in Attachment 3. The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second analyst. The data review checklist is used to document the reviews and once it has been completed, initialed and dated it must be filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file organized by instrument and date. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.8.

15.5 Reporting The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and all information required by the reference method and the laboratory quality control program.

In addition to sample results, the APH data report must include the following items:

- Method Blank results
- LCS results

- Sample duplicate results
- Internal standard results (areas) for all field samples and QC samples

15.5.1 Analysis Observations / Case Narrative Summary Form This form, which is included in the *SOP for Sample Analysis, Storage and Tracking*, must be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. This form is necessary as a means for documentation and will be reviewed along with other information when compiling the final report and case narrative.

Any sample flow controller that does not meet the post calibration check criteria (refer to Section 8.2) must be noted on this form so that it may be reported to the client.

All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

15.5.2 Significant Modifications "Significant Modifications" to the APH Method shall include, but are not limited to, any of the following and must be reported accordingly, if they occur.

(1) The use of sample collection devices other than evacuated passivated stainless steel canisters or glass Bottle Vacs (i.e., Tedlar bags).
(2) The use of alternative detectors other than GC/MS to quantify Target APH Analytes and/or hydrocarbon range concentrations.
(3) The use of extracted ions other than 120 and 134 to quantify C9-C10 aromatic hydrocarbons.
(4) The failure to provide all of the data and information required in the report form presented in Appendix 3.

Data produced using an analytical method incorporating any of the "Significant Modifications" described above may *not* be reported as APH data. APH range concentrations are method-defined parameters and as such may only be reported as APH data when produced using the method without "Significant Modifications."

Helium Pressurization – If a canister is pressurized with helium, a correction factor is applied to sample volumes extracted from the canister via auto sampler. This is due to the difference in thermal properties between helium and air. A correction factor worksheet has been generated to determine the exact volume taken from a canister and may be found at J:\\A-GCMS\Helium Pressurization (save the job as P1_____h.xls). Print the sheet and include with the data. Refer to the instruction page in the template for all of the instructions and calculations including backfilled canisters.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 This section of the standard operating procedure contains technical acceptance criteria. To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.2 Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV. Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action,* where appropriate.

16.3 Analytical Sequence Refer to Section 12.2.1 for the analytical sequence requirements. All analytical sequences and data must be recorded in an instrument run logbook.

16.4 Minimum Instrument QC (Additional) The following are additional requirements or are reiterated from previous sections.

- Internal standards used must be adequately resolved from individual compounds in the APH Component Standard.
- Retention time windows and relative retention times must be established for each target APH analyte, range "marker" compound, and internal standard initially and each time a new GC column is installed, and must be verified and/or adjusted on a daily basis (see Section 16.6.4).

16.5 Initial and Periodic Method QC Demonstrations The following procedures must be conducted as an initial demonstration of laboratory capability (IDLC). Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method or operational problems.

16.5.1 Accuracy and Precision To demonstrate initial laboratory capability, analyze a minimum of four replicate samples obtained from a humidified canister fortified with each Target APH Analyte.

- Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte and hydrocarbon range, and the replicate precision (as %RSD) of the measurements for each analyte.
- For each analyte and hydrocarbon range, the mean accuracy, expressed as a percentage of the true value, must be between 70% and 130%, and the %RSD must be less than or equal to 25. The IDLC must meet these conditions for analysis to proceed.
- If desired, the accuracy and precision evaluation may be combined with the MDL evaluation specified in Sections 16.5.2 and 16.5.3.

16.5.2 Method Detection Limits for Target APH Analytes Although the method does not require that MDL studies be performed, the APH target compounds are a subset of the EPA TO-15 analysis for which laboratory performs annual MDL determinations as follows. Analyze a minimum of seven replicate samples obtained from a canister fortified with all Target APH Analytes of interest at 3 to 5 times the calculated or estimated Instrument Detection Limits (IDLs) or at the low level initial calibration standard concentration. Analyze each replicate according to the procedures described in this document. Calculate the Method Detection Limit (MDL) of each analyte using Equations 9 and 10 and Table 6 below.

Equation 9: Standard Deviation

$$SD = \sqrt{\sum_{i=1}^{N} \frac{\left(C_i - \overline{C}\right)^2}{N-1}}$$

where:

C_i are the individual concentrations from each MDL replicate analysis

$\overline{C}$ Average (or Mean) concentration of all MDL replicate analyses

N total number of MDL replicate analyses

Equation 10: Method Detection Limit

$$MDL = (t) \times (SD)$$

where:

t = student t value at the 99% confidence level.
SD = standard deviation of the replicate analysis.

Table 6
Student t Values

Number of replicates	t value
7	3.143
8	2.998
9	2.896
10	2.821

16.5.3 Method Detection Limits for Hydrocarbon Ranges The method does not require that MDL studies be performed. However, the laboratory may choose to perform them in anticipation of client requests. Analyze a minimum of seven replicate samples obtained from a humidified canister fortified with all of the APH range calibration compounds at 3 to 5 times the calculated or estimated Instrument Detection Limit (IDL) or at the low level initial calibration standard concentration. Analyze each replicate according to the procedures described in this document. Calculate the Method Detection Limit (MDL) of each range using Equations 9 and 10 and Table 6.

16.5.4 Method Reporting Limits

16.5.4.1 Target APH Analytes The method reporting limit for each target APH analyte must be at or above the low level calibration standard and should be verified on an annual basis by analyzing at least 4 replicate samples from a canister spiked at the reporting limit, where the precision is demonstrated to be equal to or less than 25% RSD, and the mean accuracy is demonstrated to be between 70%-130% of the spiked value.

16.5.4.2 Collective Hydrocarbon Ranges The method recommends that the MRL for each hydrocarbon range be based upon the concentration of the lowest range calibration standard for the components that make up this range. The minimum MRL for each range is equal to the sum of the mass amounts of all the individual components in the lowest calibration standard point that are used for creating that range's RRF. In practice, this leads to MRLs that are so low that chromatographic

baseline noise often yields a false positive result. The laboratory will set the MRLs at or above this level so long as it meets the data quality objectives of the data user.

16.6 Ongoing Method QC Demonstrations

16.6.1 Instrument Performance Check

Acceptance Criteria – The GC/MS system must meet the mass spectra ion abundance criteria listed in Table 7. The appropriate corrective action is described below. Results of the BFB tune check as well as any actual tuning must be recorded and a copy of the tune report maintained on file.

Table 7 BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	8.0 – 40.0 percent of the base peak
75	30.0 – 66.0 percent of the base peak
95	base peak, 100 percent relative abundance
96	5.0 – 9.0 percent of the base peak
173	less than 2.0 percent of mass 174
174	50.0 to 120.0 percent of the base peak
175	4.0 – 9.0 percent of mass 174
176	greater than 93.0 percent but less than 101.0 percent of mass 174
177	5.0 – 9.0 percent of mass 176

Corrective Action – Re-analyze the BFB compound or perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria.

16.6.2 Initial Instrument Calibration

Acceptance Criteria

- Refer to Section 11.1.5 for the initial calibration procedure requirements (i.e., number of points, dropping points, etc.)
- The calculated percent relative standard deviation (%RSD, linear or quadratic regression is not allowed) for the relative response factors (RRF) for each compound in the calibration standard must be ≤30% with *Naphthalene* up to ≤40%.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable initial calibration verification (ICV) (refer to Section 16.6.3).

Corrective Action – Follow the initial calibration guidelines detailed in this document for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards. If the criteria are not met it may be necessary to perform maintenance, if this is the case then all calibration points must be re-analyzed.

16.6.3 Initial Calibration Verification Standard (ICV) / Laboratory Control Sample (LCS)

Acceptance Criteria – The spike recovery (%R) must be between 70%-130%.

Corrective Action – If the technical acceptance criteria are not met, reanalyze and if it still fails prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. A demonstration of an acceptable ICV is required.

16.6.4 Continuing Calibration Verification (CCV)

Acceptance Criteria

- The percent difference (%D) must be ≤30% (single analyte or hydrocarbon range). If more than one compound fails to meet this criteria, or any one analyte or range is >50% then the CCV is considered unacceptable.
- The relative retention time (RRT) and RRT window for each target APH analyte, internal standard, and hydrocarbon range "marker" compound must be verified with each CCV analyzed.

Corrective Action – If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it still fails prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects may be reported with the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative. Otherwise the sample affected by the unacceptable CCV shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

16.6.5 Method Blank

Acceptance Criteria – The method blank result for any target analyte must not be greater than the reporting limit and should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.

Corrective Action – If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results

are acceptable.

If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). Regardless, appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 18.3.

16.6.6 Laboratory (Sample) Duplicate

Acceptance Criteria – The relative percent difference (RPD) must be <30% when the results are >5x the MRL.

- If the RPD exceeds 30 and both results are >5x the MRL, the sample analysis must be repeated.
- If an analyte is detected in one analysis at >5x the MRL but not detected in the duplicate analysis, the analysis must be repeated.
- If an analyte is detected in one analysis at ≤5x the MRL but not detected in the duplicate analysis, the RPD is not calculable and the analysis does not have to be repeated.

Corrective Action – If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.6.7 Internal Standards

Acceptance Criteria – Internal standards must be adequately resolved from individual compounds in the APH calibration standard. A minimum separation requirement of 50% (maximum peak height to valley height) must be met, particularly for n-hexane and bromochloromethane (IS1). The internal standard area counts of each sample, blank, and Laboratory Control Sample must be evaluated against the corresponding continuing calibration standard or the midlevel initial calibration standard (if analyzed in the same sequence). The internal standard area counts must be within 50-200% of the continuing calibration standard area counts. If the internal standards fall outside this range, the sample, blank, or Laboratory Control Spike must be reanalyzed.

Corrective Action – If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If the problem is corrected with the lower volume analysis, report those results.

16.6.8 Sample Analysis

Acceptance Criteria – Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.

- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria.

- All target analyte peaks must be within the initial calibration range or reported with the appropriate data qualifier.
- The internal standard with each sample must comply with the requirements listed in Section 16.6.7.
- Each analyte, in order to be reported, must meet the qualitative identification requirements listed in Section 12.9.1.

Corrective Action – When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.7 Sample's Holding Time Expired The customer is to be notified that the sample's holding time was missed and the customer is to decide if the sample analysis is to continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual.

Note: No analyte results may be reported with an unacceptable initial calibration or initial calibration verification standard. However, any analyte not meeting such requirements (and the initial calibration is to be used) must be eliminated from the reporting list and any action taken fully documented.

18.2 Continuing Calibration Verification

- When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
- If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects may be reported with the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative. If this is the case then a full explanation must be noted in the case narrative of the final report. Refer to Section 15.5 for additional reporting requirements.

18.3 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

18.4 Laboratory Control Sample All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

18.5 Laboratory Duplicate All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

18.6 Internal Standard All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

18.7 Estimated Sample Results

18.7.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this standard operating procedure.

18.7.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

18.7.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported has having less certainty by reporting with the appropriate data qualifier.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use.

19.2 Method Detection Limit (MDL) This method does not require that MDL studies be performed. However, the APH target compounds are a subset of the EPA TO-15 analysis for which the laboratory performs MDL studies. The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 of the *SOP for Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS)* and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

19.3 Accuracy and Precision Refer to Section 16.5.1 above for information on replicate precision and accuracy criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets

the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the TO-15 Method Manual.

19.4 Selectivity Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 98 or newer version
- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

19.5 Demonstration of Capability

See Sections 16.5 and 16.6 for initial and ongoing method QC requirements.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
09.0	03/21/15	C. Humphrey	Section 4 – Revised section to include Hazard Assessment table
			Section 12.9 – Added Note 2
			Attachment 3 – Replaced Data Review Checklist with combined TO-15/MAPH Daily QC and Sample Review Checklists

21) References and Related Documents

21.1 *Method for the Determination of Air-Phase Petroleum Hydrocarbons (APH)*, Final Revision 1, Massachusetts Department of Environmental Protection, December 2009.

21.2 *SOP for Batches and Sequences*, SOP ID ADM-BATCH_SEQ

21.3 *SOP for Making Entries onto Analytical Records*, SOP ID CE-QA007

21.4 *SOP for the Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*, SOP ID CE-QA011

21.5 *SOP for Manual Integration Policy*, SOP ID CE-QA002

21.6 *SOP for Nonconformance and Corrective Action*, SOP ID CE-QA008

21.7 *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters,* SOP ID SMO-CanCert

21.8 *SOP for Flow Controllers and Critical Orifices,* SOP ID SMO-Flow_Cntrl.

21.9 *SOP for Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS),* SOP ID VOA-TO15

21.10 2009 TNI Standards

22) Appendix

22.1 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Daily QC and Sample Review Checklists

Attachment 1

Training Plan

Training Plan for Analysis of Air-Phase Petroleum Hydrocarbons (APH) by GC/MS

SOP Title: _____ Revision:_____ Date: _____

Trainee: _____ Trainer: _____ Instrument: _____

1. Read SOP — Trainer ____ Trainee ____ Date _____

2. Read Method — Trainer ____ Trainee ____ Date _____

3. Demonstrated understanding of the scientific basis of the analysis — Trainer ____ Trainee ____ Date _____
   Training Duration _____
   - Whole air sample preconcentration techniques
   - Gas chromatography
   - Mass spectrometry

4. Demonstrated familiarity with related SOPs — Trainer ____ Trainee ____ Date _____
   Training Duration _____
   - SOP for Batches and Sequences
   - SOP for Making Entries onto Analytical Records
   - SOP for Manual Integration Policy
   - SOP for Significant Figures
   - SOP for Nonconformance and Corrective Action
   - SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation
   - SOP for Cleaning and Certification of Summa Canisters

5. Observe performance of SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   - ___sample preparation/dilution and sample loading and analysis
   - ___analytical sequence setup
   - ___standard preparation
   - ___BFB tuning evaluation/initial calibration/initial calibration verification
   - ___continuing calibration verification
   - ___EnviroQuant introduction
   - ___data reduction and reporting
   - ___canister handling

6. Perform SOP with supervision *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   - ___sample preparation/dilution and sample loading
   - ___analytical sequence setup
   - ___standard preparation
   - ___BFB tuning evaluation/initial calibration/initial calibration verification
   - ___continuing calibration verification
   - ___sample analysis
   - ___EnviroQuant use
   - ___data reduction and reporting
   - ___canister handling

7. Independent performance of the SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   - ___sample loading and sample dilutions
   - ___analytical sequence setup
   - ___standard preparation
   - ___BFB tuning evaluation/initial calibration/initial calibration verification
   - ___continuing calibration verification
   - ___sample analysis
   - ___EnviroQuant proficiency
   - ___data reduction and reporting
   - ___canister handling
   - ___initial demonstration of competency
     - -4 Laboratory Control Samples

8. Instrument operation and maintenance *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   - ___autosample
   - ___GC and capillary column installation
   - ___mass spectrometer
   - ___data system

Attachment 2

Initial Calibration Checklist

Method: **MAPH**
ICAL Date: _____
Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS____

Air-Phase Petroleum Hydrocarbons
Initial Calibration Review Checklist

Analyst **Reviewer**

☐ 1. Is the required documentation in the ICAL file? ☐
- ☐ BFB Tune analysis Report ☐
- ☐ Calibration Status Report (aka Calibration History) ☐
- ☐ Response Factor Report/Percent RSD (target analytes) ☐
- ☐ Percent RSD Report (hydrocarbon ranges) ☐
- ☐ Quantitation Report for each calibration standard (including manual integration documentation) ☐
- ☐ ICV Quantitation Report ☐

☐ 2. Was the ICAL performed continuously (i.e., not interrupted for maintenance or for sample analysis)? ☐

☐ 3. Have all the calibration standards been analyzed within 24 hours of each other? ☐

☐ 4. Does the BFB tune check standard analysis at the start meet the tune criteria? ☐

☐ 5. Are all the analytes in the blank analysis <MRL? ☐

☐ 6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels? ☐

☐ 7. Were the standards analyzed from low concentration to high concentration? ☐

☐ 8. For each analyte or range, are there no levels skipped? ☐

☐ 9. For each analyte or range, is there only one value used for each calibration level? ☐

☐ 10. For each analyte range, is the lowest standard's concentration at or below the analyte's MRL? ☐

☐ 11. If a calibration level is dropped, are all the responses for each target analyte and range dropped and is the information noted in the ICAL explaining the reason? ☐

☐ 12. Is the average RSD ≤30% for all analytes and ranges, except *naphthalene* can be ≤40%? ☐

☐ 13. For the ICV analysis, are all the analytes within 70%-130% recovery? ☐

☐ 14. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages. ☐

COMMENTS:

Analyst: _____ Date: _____

Secondary Reviewer: _____ Date: _____

Attachment 3

Daily QC and Sample Review Checklists

Daily QC Review Checklist

(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

EPA Compendium Method TO-15

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		**Reviewer**
☐	1. Is the required documentation present? ☐ CORRECT BFB Tune analysis Report ☐ CCV analysis Quantitation Report & %D Report ☐ LCS analysis Quantitation Report ☐ MB analysis Quantitation Report	☐
☐	2. BFB **tune** check standard analysis meet the tune criteria for the method indicated above?	☐
☐	3. Analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**?	☐
☐	4. Does the **CCV** have a difference ≤30% for all analytes? [Note all outliers biased high and/or low]	☐
☐	5. All **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)?	☐
☐	6. All **IS** responses within ±40% of CCV or the midpoint in the ICAL?	☐
☐	7. All **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%)	☐
☐	8. All analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)?	☐
☐	9. **LCS** %R within the lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)?	☐
☐	10. All analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits?	☐

COMMENTS:

Air-Phase Petroleum Hydrocarbons

☐	1. Does the **CCV** meet the following criteria? • Percent difference ≤30%. • One compound or range can be >30%, but less than 50%. • No single analyte or range may be >50%. [Note outliers biased high and/or low]	☐
☐	2. Does lab **duplicate** meet an RPD of ≤30% for results >5x MRL? Repeat analysis if:	☐
☐	3. Are the analytes in the **LCS** within 70%-130% recovery?	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐ LIMS Run Approval ☐ LIMS Supervisor Approval

Analyst: _____ Secondary Reviewer: _____

Date: _____ Date: _____

Sample Review Checklist
(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

EPA Compendium Method TO-15

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____ **Project #:** _____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. All analyte hits in the samples within the **calibration range** and/or noted?	☐
☐	2. All **peak integrations** acceptable?	☐
☐	3. All **manual integrations** flagged and documented?	☐
☐	4. Have **Q values** been verified for each peak?	☐
☐	6. All **calculations** correct?	☐
☐	7. Has the analyst initialed and dated each **quantitation report**?	☐
☐	8. For **TICs** are the relative intensity and other requirements met?	☐
☐	9. **Auto report** correct?	☐
☐	10. **MRL** = _____ ☐ ng ☐ pg (ethanol, acetone, vinyl acetate = 5.0ng)	☐
☐	11. Pressurized with **Helium**? Is the worksheet completed for all samples?	☐
☐	12. Report to **MDL**? ☐ Yes ☐ No	☐
☐	13. **Global Minimum Detection Limit** = _____ ☐ ng ☐ pg	☐
☐	14. **DOD:** Are **manual integrations** notated in the **case narrative?**	☐

COMMENTS:

Air-Phase Petroleum Hydrocarbon

☐	1. Are all manual **integrations** flagged and documented (except for HC ranges)?	☐
☐	2. Are all peak **integrations** acceptable?	☐
☐	3. Has the analyst initialed and dated each **quantitation report**?	☐
☐	4. Are the associated ICAL responses correct?	☐
☐	5. Are the sample responses entered into the template correctly?	☐
☐	6. Are the TO-15 target compounds entered into the template correctly?	☐
☐	7. Does the lab **duplicate** meet a RPD of ≤30% for results >5x the MRL? Otherwise, repeat analyses if:	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐ LIMS Run Approval

Analyst: _____

Date: _____

☐ LIMS Supervisor Approval

Secondary Reviewer: _____

Date: _____

ALS Standard Operating Procedure

Proprietary - Uncontroll Copy

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCED METHOD:	EPA TO-15
SOP ID:	VOA-TO15
REV. NUMBER:	22.0
EFFECTIVE DATE:	03/21/2015

STANDARD OPERATING PROCEDURE

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

EPA TO-15

SOP ID:	VOA-TO15	Rev. Number:	22.0	Effective Date:	03/21/2015

Approved By: Wida Ang — Date: 3/13/15
Team Leader (VOA GC/MS) - Wida Ang

Approved By: Chris Parnell — Date: 3/13/15
Technical Manager (VOA GC/MS) - Chris Parnell

Approved By: Chaney Humphrey — Date: 3/13/15
QA Manager - Chaney Humphrey

Approved By: Kelly Horiuchi — Date: 3/13/15
Laboratory Director - Kelly Horiuchi

Archival Date: _____ Doc Control ID#: Non-Controlled Editor: _____

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DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1) Scope and Applicability

1.1 This procedure is based on and incorporates the requirements detailed in EPA Compendium Methods TO-15 and TO-14A and is used to quantify a wide range of volatile organic compounds (VOCs) in gaseous matrices collected in gas collection bags (method modification) and specially prepared stainless steel canisters or glass bottles. This method typically applies to ambient concentrations of VOCs 0.50ug/m3 (down to 0.10ug/m3 for low level ambient analyses) and above for the SCAN mode and 0.010ug/m3 and above for the SIM mode; however, refer to Tables 3 and 3A for the specific laboratory initial calibration ranges for each target compound. The method requires VOC enrichment by concentrating up to one liter of a sample volume, with a virtually unlimited upper concentration range using dilutions from source level samples.

In this document, Tables 2 and 2A (see Note 1 below) list compounds that can be determined by this procedure along with their corresponding laboratory method reporting limits (MRLs) and method detection limits (MDLs). The reported MRL may be adjusted higher; however, the capability of achieving lower MRLs for specific project requirements must be thoroughly demonstrated (by an acceptable initial calibration and method reporting limit check standard) and documented as long as the MRL is higher than the current method detection limit for each compound. Additional compounds may be analyzed according to this procedure as described in the referenced methods as long as the requirements of this document are adhered to; however, if a compound is not listed in the TO-15 method, refer to Note 1 below. The number of samples that may be analyzed in a 24-hour period is about twenty. The number of sample results that may be reduced in an eight-hour day is approximately twenty.

2) Summary of Procedure

2.1 The analytical method involves using a high-resolution gas chromatograph (GC) coupled to a mass spectrometer (MS). The GC/MS utilizes a linear quadrupole system, which allows for it to be operated by either continuously scanning a wide range of mass to charge ratios (SCAN mode) or by Select Ion Monitoring mode (SIM), which consists of monitoring a small number of ions from a specified compound list.

An aliquot of an air sample is concentrated on a solid adsorbent trap (either cryogenically or fan cooled glass beads or stronger adsorbents at higher temperatures) to collect the analytes of interest. To remove co-collected water vapor, the concentrated sample then goes through a water removal (dry purge) step. After the sample is pre-concentrated on a trap, the trap is heated and the VOCs are thermally desorbed onto a refocusing cold trap. The VOCs are then thermally desorbed onto the head of a capillary column once the cold trap is heated. The oven temperature (programmed) increases and the VOCs elute and are detected by the mass spectrometer.

Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the

system response to the primary fragment for known amounts of the compound. This method utilizes the internal standard calibration technique; refer to Section 3.16 for a complete definition.

3) Definitions

3.1 Cryogen A refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Liquid nitrogen (cryogen) is used for this purpose and it has a boiling point of -195.8°C.

3.2 Gauge Pressure Pressure measure with reference to the surrounding atmospheric (barometric) pressure, usually expressed in units of psig. Zero gauge pressure is equal to atmospheric pressure.

3.3 MS-SCAN Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

3.4 MS-SIM Mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].

3.5 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.6 Neat Stock Standard A purchased, single component assayed reference material having a stated purity used to prepare working calibration standards.

3.7 Stock Standards Solution A concentrated solution of one or more target analytes at a known concentration purchased from a reputable commercial vendor. Stock standard solutions are used to prepare working calibration standards.

3.8 Intermediate Calibration Standard A solution of one or more target analytes at a known concentration prepared either from one or more neat stock standards or from one or more stock standards solutions.

3.9 Working Calibration Standard A solution of all the target analytes at a known concentration prepared either from one or more intermediate calibration standards and/or from one or more stock standard solutions.

3.10 Calibration or Standard Curve A calibration or standard curve is a graph which plots the concentration of a compound (or an analyte) versus the instrument response to the compound.

3.11 Initial Calibration Verification (ICV) Standard A solution prepared in the laboratory containing known concentration(s) of analytes of interest. The solution is prepared from neat stock standards and/or stock standards solutions which are from a different source than the standards used to prepare the working calibration standards.

3.12 Continuing Calibration Verification (CCV) Standard A working calibration standard which is analyzed at specific intervals in order to verify that the instrument continues to meet the calibration criteria.

3.13 Field Sample A sample collected and delivered to the laboratory for analysis.

3.14 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop "ticks" have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.15 Batch Quality Control (QC) Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD).

3.16 Internal Standard Calibration Compares the instrument responses from the target compound in the sample to the responses of specific standards (called internal standards), which are added to the sample or sample preparation prior to analysis. The ratio of the peak area (or height) of the target compound in the sample or sample preparation is compared to a similar ratio derived for each calibration standard.

3.17 May This action, activity, or procedural step is neither required nor prohibited.

3.18 Must This action, activity, or procedural step is required.

3.19 Shall This action, activity, or procedural step is required.

3.20 Should This action, activity, or procedural step is suggested, but not required.

3.21 SOP Standard Operating Procedure

3.22 Service Request A form generated, at the time of sample receipt, which details pertinent information such as client name, address, contact, client and laboratory sample identifications, sampling and receipt dates and times, requested analyses, sample type, canister pressures (initial and final), and the service request number (unique number for each submitted job) and serves as an inter-laboratory "custody" form which accompanies all samples throughout the laboratory.

3.23 Selectivity Selectivity of a method refers to the extent to which it can determine particular analyte(s) in a complex mixture without interference from other components in a mixture. Another definition is the extent to which a particular method can be used to determine analytes under given conditions in the presence of other components of similar behavior.

3.24 Limit of Detection (LOD) The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DoD Clarification). For consistency purposes, the LOD may be referred to as the MDL once it is reported; however, full verification will be on file in the laboratory per the procedures detailed in this document.

3.25 Limit of Quantitation (LOQ) The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. (DoD Clarification). For consistency purposes and since the LOQ and MRL are equivalent with regards to laboratory procedure, the LOQ will be referred to as the MRL in this document and once it is reported. Full verification will be on file in the laboratory per the procedures detailed in the document.

3.26 Detection Limit (DL) / Method Detection Limit (MDL) The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type 1 error) is 1%. (DoD Clarification). For consistency purposes, the DL may be referred to as MDL. Also, as far as reporting is concerned the MDL will be raised up (where necessary) to the verified LOD per the procedures defined in this document and reported accordingly.

4) Health and Safety Warnings

4.1 Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, Safety Data Sheet (SDS) location, and the laboratory waste management plan for the safe disposal of chemicals and samples.

4.2 Pollution Prevention and Waste Management

All waste disposals shall be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal.* In addition, canisters must be cleaned in accordance with the requirements detailed in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters.*

4.3 This procedure may include CHEMICAL, OPERATIONAL and/or EQUIPMENT hazards. Employees must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1: Standard and Sample Preparation	**Hazards**	**Preventative Measures**
Compounds, mixtures of compounds, standards, surrogates, and samples.	Exposure to potential health hazards through absorption through skin. Inhalation hazards.	Reduce exposure through the use of gloves and fume hoods. Safety glasses must be worn when working in the prep lab. Care should be taken when handling standard material in a neat or highly concentrated form. Personal protective clothing (safety glasses, gloves, and lab coat) are required when handling standard material in neat form. Consult Safety Data Sheets (SDS) for compounds being handled in this procedure, and be familiar with proper safety precautions.
Job Task #2: Working with Liquid Nitrogen	**Hazards**	**Preventative Measures**
Turning valves and handling tubing and fittings that have been in contact with the cryogen.	Can cause serious tissue damage (frostbite) with only a few seconds of contact.	Wear neoprene or leather gloves. Valves on cryogen dewars should be opened slowly so leaky fitting can be identified.
Job Task #3: Working with Pressurized Gases	**Hazards**	**Preventative Measures**
Using and moving compressed gas cylinders.	Gas leak, fire, and explosion. Personal injury due to falling during transport.	All cylinders must be secured in an upright position to a wall or immovable counter with a chain or a cylinder clamp when not in use. Keep safety caps on when cylinders are not in use. A handcart must be used when transporting cylinders. The cylinder must be secured to the handcart with a chain or belt. The regulator should never remain on small "D" size cylinders following use. Full cylinders must be kept separate from empty cylinders. Flammable gases (i.e. pressurized hydrogen) must be clearly labeled. Flammables and oxidizers must be separated by a ½-hour fire wall or by at least twenty feet.
Job Task #4: Glass Syringes	**Hazards**	**Preventative Measures**
Glass syringe use	Skin lacerations and punctures.	The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

5) Cautions

5.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

The instrument maintenance log must be kept current. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The entry in the log must include.

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful tune and continuing calibration or initial calibration and the file number that accompanies the data will serve as proof that the maintenance is complete and the instrument is in working order.

The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, changing the pump oil, cleaning the source, ordering a part. In addition, a notation should be made in the logbook stating that no samples were analyzed during the days that the instrument was down and no active maintenance was being conducted (i.e., where no other notation was made in the logbook for those days).

5.2 Concentrating Trap

Routine maintenance includes periodic solvent cleaning of the Silco steel lines in the valve oven if contamination is suspected. Also, periodic replacement of the multi-sorbent or partial replacement of the trap if analyte specific deterioration is detected is required. For specific trap information refer to the instrument maintenance logbook and electronic method manual.

After repacking, the trap should be baked at 265°C for a minimum of two hours (or until a clean blank is generated) and a partial repacking requires baking (at 265°C) the trap for a minimum of 20 minutes (or until a clean blank is generated).

5.3 GC System

Column performance is monitored by observing both peak shapes and column bleed. Over time, the column will exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced (see Section 9.5). Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Declining performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.4 Mass Spectrometer

The Mass Selective Detector (MSD) ion source requires periodic cleaning to maintain proper performance. Symptoms of a dirty ion source include difficulty keeping the MSD in tune and fluctuating internal standard areas. The vacuum system should be serviced every six months, including changing the pump oil and checking the molecular sieve in the back-streaming trap.

5.5 Instrument Tuning

The instrument is tuned with guidance from the procedure described in the HP Operations Manual, when necessary.

5.6 Computer Troubleshooting

Computer care and troubleshooting is conducted by the IT department. Refer to Section 9.6 for the computer hardware and software requirements.

Computers are selected to meet or exceed operating system and or acquisition software requirements. Periodic upgrades of memory are performed to maintain or improve system performance and reliability. Upgrades may be performed on systems until instrument hardware configurations become the limiting factor.

Basic Troubleshooting Outline:

1) Document occurrence and severity in IT Log
2) Interview user(s)
3) Investigate any available logs (Event Logs, Acquisition Logs, etc.)
4) Determine if problem is isolated (single user or acquisition) or widespread (multi user or network).
5) If multiple possibilities exist for cause, then eliminate in systematic manner.
6) Hardware issues are addressed with component replacement (beginning with most suspect portion).
7) Software issues are addressed first with internet investigation (user blogs, software source updates/findings).
8) Network issues are investigated from the Server, to Switch, to Network Card; utilizing all available managed devices to help discover possible failure points.
9) In some cases, system corruption may require reload or complete system replacement.
10) Finalize documentation in IT Log with actions taken
11) Perform periodic follow-up with User and review any log found to have suspect events that suggested source of issue.

6) Interferences

6.1 Summa Canisters

Canisters shall be stored in a contaminant free location and shall be capped tightly during shipment to prevent leakage and minimize any compromise of the sample. The pressure/vacuum is checked prior to shipment and upon receipt from the field. Any problems with the sample from the field are noted and the Project Manager contacted.

Also, canisters must be cleaned and certified to be free from target analytes before being shipped to the field for sample collection. The procedure is described in detail in the *SOP for Cleaning and Certification of Summa Canister and Other Specially*

Prepared Canisters (refer to this procedure as well as Section 16.7 for the acceptance criteria).

Current laboratory practice entails the segregation of 6L canisters into ambient (low) level and source levels. All the ambient canisters are used for low level (indoor air, ambient air) projects and not intentionally for soil gas, SVE monitoring, or other higher level applications. It may be necessary to "retire" an ambient canister and re-assign for source level use if high concentrations are encountered. This decision will be made by management based on analytical concentrations and what compounds were encountered at these levels. If the level of any analyte is detected above 5,000ug/m*3* in the ambient can, then the supervisor/team leader must be contacted to determine if the canister(s) is to be retired. If retirement is decided upon, make a notation on the sample tag (or other color coded tag) of each canister in question. The notation must contain the analyte, threshold levels and retirement from ambient use (initial and date notation) so that the canister conditioning/management department may properly execute the retirement.

6.2 Analytical System

The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with buna-N rubber components must be avoided.

6.3 Carbon Dioxide

Excessive levels of carbon dioxide present in a sample may interfere with analysis by freezing up the cryogenic trap. A smaller aliquot must be analyzed to eliminate this problem, or the sample should be analyzed using the higher temperature multi-adsorbent trapping technique which allows carbon dioxide to pass.

6.4 Gas Collection Bags

This procedure covers the use of gas collection vessels such as Tedlar® or Mylar® bags. However, due to the nature of these types of bags it is not recommended that clients use this option for ambient air samples. Sample collection bags made out of ®Tedlar have contaminants that are inherent to the manufacturing process. The two main contaminants are phenol and N,N-Dimethylacetamide. However, this only becomes a problem when the concentration levels in the sample are low ppbv such as ambient air monitoring samples where more of the sample usually has to be concentrated and analyzed. To minimize the loss of sample integrity, a 72-hour hold time has been incorporated into the procedure.

6.5 Glassware

Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware results in discrete artifacts and/or elevated baselines in the detector profiles should be minimized. All glassware associated with this method must be scrupulously cleaned to avoid possible contamination. The cleaning shall be performed in accordance with the procedure outlined in the *SOP for Glassware Cleaning*. The use of high purity water, reagents, and solvents helps to minimize these problems.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP

may perform analysis, interpretation and peer review of the results. Data reduction and/or peer review may be performed by another qualified employee. This employee must be familiar with the analytical technique and have completed a data review training plan to ensure familiarity with specific analysis and requirements.

7.2 The supervisor/manager must ensure that method proficiency is documented initially and whenever significant changes in the instrument type, personnel, and matrix or test method are made.

7.3 The department supervisor/manager or designee shall perform final review and sign-off of the data.

7.4 Demonstration of Capability

All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy*. Demonstrations shall also be performed in accordance with the 2009 TNI Standards (Volume 1 Module 4 Section 1.6) and DoD Quality Systems Manual 5.0. Attachment 1 shall be used to document the training plan for new analysts' initial demonstration. Additionally, these demonstrations are performed anytime there is a change in instrument type, personnel or method.

Once performance is found to be acceptable, a required certification statement must be completed by the QA Manager and either the immediate supervisor or Laboratory Manager and retained on file as a demonstration of compliance.

7.4.1 Quarterly Demonstration A demonstration of method sensitivity must be performed *quarterly on each instrument* performing this method.

1) A spike at the current LOD must be analyzed.

2) Verification of precision and bias at the LOQ must be performed.

Refer to Section 11.1.4.2 (LOQ) and 12.14.1 (LOD) for additional information on how these demonstrations are to be performed as well as the acceptance criteria.

7.4.2 Annual Demonstration Each analyst must perform this demonstration both initially and annually. Analyze four LCS standards at 1-4x the MRL (LOQ) either concurrently or over a period of days as a verification of precision and bias of the quantitation range. The standard deviation (n-1) and average percent recovery of the four replicates are compared against the method requirement for precision (±25%) and current laboratory control limits for bias/LCS.

7.4.3 Change in Personnel, Instruments, Method and/or Matrix The requirements in Sections 7.4.1 and 7.4.2 must be performed per the schedule noted and when there is a change in personnel, instruments, method or matrix. "Change" refers to any change in personnel, instrument, test method, or sample matrix that potentially affects the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, or other components of the sample analytical system, or a method revision).

All completed attempts at this demonstration must be completed and turned into the QA department for retention.

8) Sample Collection, Handling, and Preservation

8.1 Air samples are collected in the field and delivered to the laboratory and shall be collected in either a specially prepared, leak-free, stainless steel pressure vessel (with valve) of desired volume (e.g., 6L), a glass sampling bottle (Bottle Vac, Entech

Inntruments) or a sample collection bag (Tedlar). Canister samples may either be grab or time integrated (using a variable flow controller, refer to the *SOP for Flow Controllers and Critical Orifices*) utilizing the canister vacuum to draw the sample. Bags require the use of an upstream pump or a "lung machine."

8.2 There are no special preservation requirements for either canisters, Bottle Vacs or bags. However, bags should be stored in an environment free from puncture or deterioration sources (by hanging them from clips), labeled with the specific service request number, in accordance with the *SOP for Laboratory Storage, Analysis and Tracking*. Canisters and bottles should be stored on the appropriate shelves until they are to be analyzed.

8.3 Sample collection bags must be analyzed within 72 hours from the confirmed time of sampling. Samples received by the laboratory shall be analyzed within 30 days of sampling or sooner if project specific requirements dictate. Programs, which have shorter recommended or required hold times, include the Department of Toxic Substances Control (DTSC), which advises a 72 hour hold time. The Minnesota Pollutions Control Agency (MPCA) and EPA Region 9 both require a 14 days hold time. Additionally, the MPCA does not allow the use of Tedlar bags for sampling or sample dilution. The DTSC requirement is an advisory notice, but the laboratory shall make every effort to comply. However, the following statement shall be added to each report where sample analyses do not meet the 72 hour hold time and the client project is intended to comply with DTSC requirements. "The recommended 72-hour hold time for the analysis of TO-15 was exceeded per the DTSC and LARWQCB Advisory – Active Soil Gas Investigations document dated January 28, 2003; however, this specific hold time statement is advisory and not considered as regulation. In addition, the samples were analyzed within the EPA Method TO-15 stated requirement of 30 days."

9) Equipment and Supplies

9.1 Additional instruments and/or differing models may be utilized as long as they are equivalent and meet the minimum requirements of this document.

9.2 Gas Chromatograph (GC)

An instrument capable of temperature programming, with a column oven that may be cooled to sub-ambient temperature at the start of the gas chromatographic run to result in the resolution of the VOCs.

Hewlett Packard 5890 Series II Plus
Hewlett Packard 6890 Series
Hewlett Packard 6890A Series
Agilent 6890N Series
Agilent 7890A Series

9.3 Autosampler

Tekmar-Dohrmann AUTOCan Autosampler:	14-ACAN-074
Concentrating Trap (cryogenic trap, built-in):	14-6938-020
Cryofocusing Module w/split valve:	14-6520-A00
GAST Vacuum Pump:	DOA-P104-AA or equivalent

9.4 Mass Spectrometer (MS)

A MS capable of scanning from 34 to 350 amu every second or less, using 70 volts

(nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

Hewlett Packard 5972 Series
Hewlett Packard 5973 Series
Agilent 5973N
Agilent 5973 *inert*
Agilent 5975B *inert*
Agilent 5975C *inert*

9.4.1 Ionization Gauge Controller

- Agilent: 59864B
- Granville-Phillips 330 Ionization Gauge Controller: 330001/2/3
- Hewlett Packard Ionization Gauge Controller: 59864B

9.5 Analytical Column

Any analytical column capable of separating the compounds of interest may be used. The capillary column should be directly coupled to the source of the mass spectrometer. The following are suggested columns; an alternative column may be used as long as sufficient peak resolution and separation is achieved.

- Restek Rxi-1ms Fused Silica Capillary Column; 30m x 0.25mm ID 1.0µm film thickness

  OR

- Restek Rxi-1ms Fused Silica Capillary Column; 60m x 0.25mm ID 1.0µm film thickness

9.6 Data Systems

IBM-compatible PC with Windows 95/98/NT/XP/7 (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library (2002 version or newer) or equivalent.

9.7 Canister Pressurization Station

Vacuum/Pressure Gauge [0 to –30 inHg; 0-90 or 100 psig]

9.8 Canister Sampling Devices

Refer to the *SOP for Flow Controllers and Critical Orifices* for specific calibration and other pertinent information.

- VICI Condyne Model 300 Flow Controller
- Critical Orifices (Laboratory manufactured)

9.9 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent
- Lab Commerce, Stabilizer Model 22.4L, 2.4L Canisters or equivalent
- Restek Corporation, #24203, 3.0L Silco Canisters or equivalent

- Tedlar bags – 0.5L, 1L, 3L, 5L, 10L, 25L, and 40L (other sizes are available; however, the volumes that are listed encompass the majority of the bags supplied and the samples submitted to the laboratory).

9.10 Dynamic Dilution System

- Entech Dynamic Diluter Model 4620A
- Toshiba laptop computer Model 2210CDT/6.0 and Software NT460

10) Standards and Reagents

10.1 Reagents and Equipment

10.1.1 UHP Grade Helium (99.999%) (GC carrier gas, preconcentrator purge/sweep gas, pressurization gas)

10.1.2 Cryogen - Liquid nitrogen from bulk tank or 50 psig dewars (used to cool preconcentrator traps)

10.1.3 UHP/Zero Grade Air (canister pressurization)

10.1.4 ASTM Type II Water, DI water or equivalent

10.1.5 UHP Grade Nitrogen (99.999%) (additional pressurization gas, based on other methods requested – modification to method)

10.2 Standards

Standards are prepared for both SCAN and Selective Ion Monitoring (SIM) modes according to the procedures detailed in this section. The preparation of standards for the analysis of air samples is carried out by following the procedure, "Preparation of Gas Phase Standards for Ambient Air Analysis", Application Note, Spring 96, Vol. 6.5, *Tekmar*-DOHRMANN AutoCan User's Manual. Neat standards that are used for making trace gas standards must be of high purity; generally a purity of 98 percent or better is commercially available.

10.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Mixture Prepare a standard solution of p-Bromofluorobenzene (BFB-used as both a tune check and surrogate compound), bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene, 1,2-dichloroethane-d4(surrogate), and toluene-d8(surrogate) at 500µg/m³ each in humidified zero air (Section 9.2.1.2). Prepare this standard according to the procedure outlined in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note. This standard may also be prepared from a neat cocktail as in Section 10.2.2.2.1 or as stated in Section 10.2.1.3.

10.2.1.1 An intermediate standard is prepared from neat compounds in a glass static dilution bottle (SDB). After the volume of the SDB is determined, calculate the mass of each compound to be spiked to achieve a final concentration of 5.0µg/ml. Then use the density of each neat compound to calculate the microliter amount to be spiked into the SDB. The SDB is then heated for a minimum of one hour at ~60°C to completely volatilize all components.

Concentration of the intermediate standard prepared in a SDB is 5.0µg/mL. The amount required to achieve this concentration is determined through the use of the following equation.

$$A = \frac{(C)(V)}{D} \qquad \text{(Equation 1)}$$

Where:

A Amount of each compound required to achieve the desired concentration of the standard in the SDB (µL)
C Desired concentration of SDB (µg/mL)
V Actual volume of the SDB (mL)
D Density of the compound in question (µg/µL)

Example:

Calculate the amount of neat bromochloromethane needed to achieve the final concentration of 5.0µg/mL of that compound in the SDB.

V = 2010mL
D = 1934.4µg/µL
C = 5.0µg/mL

$$A = \frac{\left(5.0\frac{\mu g}{mL}\right)2010mL}{1934.4\frac{\mu g}{\mu L}} = 5.2\mu L$$

Density (µg/µL)	Compound
1934.4	Bromochloromethane
1170.1	1,4-Difluorobenzene
1157	Chlorobenzene-d5
1307	1,2-Dichloroethane-d4
943	Toluene-d8
1593	BFB

10.2.1.2 The Working standard is prepared in a Summa canister by spiking an aliquot of the stock SDB standard (Section 10.2.1.1) using a heated gastight syringe. Connect a cleaned, evacuated Summa canister to a source of pure diluent gas (humidified zero air) using a Teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum. Spike the SDB stock and following removal of syringe a small flow of diluent gas to flush the spike into the can. Pressurize the can to positive 83.3 psig with humid zero air, and allow the contents to equilibrate for approximately 24 hours before using.

Concentration of the working standard prepared in a Summa canister is 500ng/L. The final pressure of the canister is 83.3psig; therefore, the pressurized volume is 40L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 2)

Where:

PV Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PF = $\frac{P_{atm}+P_f}{P_{atm}+P_i}$

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister at 1atm

P_{atm} Atmospheric Pressure = 14.7psig

Example:

$\frac{14.7+83.3}{14.7+0}(6L)$ = 40L

In order to prepare the canister with a concentration of 500ng/L, it must be determined how much of the intermediate standard is required. This is achieved through the use of the following equation.

$$A = \frac{(F)(V)}{(C)\left(1000\frac{ng}{\mu g}\right)} \qquad \text{(Equation 3)}$$

Where:

F Desired concentration of working standard (ng/L)

V Pressurized Volume of Canister (L)

C Concentration of prepared SDB (µg/mL)

A Amount of standard (mL) of the SDB required to obtain the desired working standard concentration

Example:

$$A = \frac{500\frac{ng}{L}(40L)}{\left(5.0\frac{\mu g}{mL}\right)\left(1000\frac{ng}{\mu g}\right)} = 4mL$$

10.2.1.3 Currently the working standard is purchased in a cylinder at a certified concentration of 500ng/L (prepared by Linde SPECTRA Environmental Gases, Alpha, NJ).

10.2.1.3.1 For SCAN analyses, the working standard is filled directly into a summa canister to a pressure of 70 to 80 psig.

10.2.1.3.2 For SIM analyses, the working standard is diluted and pressurized with humid zero air to the desired concentration

using Equation 2 in Section 10.2.1.2. Typical concentrations will be 20ng/L, 40ng/L or 50ng/L.

10.2.2 Initial Calibration (ICAL) Standard Prepare the primary source calibration standards in Summa canisters with nominal concentrations of 1ng/L (optional), 20ng/L and 200ng/L for analyses in SCAN mode and 0.1ng/L, 5.0ng/L, and 200ng/L for analyses in Selective Ion Monitoring (SIM) mode for each of the target analytes. Differing injection volumes will create the standard concentrations listed in Tables 3 (SCAN) and 3A (SIM) of this document. The full list of analytes which are analyzed according to this method can also be found in Tables 2 (SCAN) and 2A (SIM).

Standards are prepared by diluting the stock standard with humid zero air into a Summa canister. The stock standard is a certified custom-blended cylinder (prepared by Linde SPECTRA Environmental Gases, Alpha, NJ). Refer to Tables 3 and 3A for the list of analytes and certified concentrations in the purchased cylinder.

10.2.2.1 Working standards are prepared into Summa canisters using the Entech Dynamic Diluter. Turn on the power to the diluter one hour prior to using to allow for the components to come to thermal equilibrium. Connect the computer and start the software. Connect a Zero Air source to the humidification chamber (flow controller #1). Connect stock standard cylinder#1 to flow controller #2 inlet. Open the cylinder valves. Adjust the inlet pressures to 50 to 60psig.

Standard Concentration Selection: The concentration of the three working standards prepared in Summa canisters should be 200ng/L, 20ng/L and 1ng/L (depending on the dynamic range of the initial calibration include 1ng/L if a 0.08ng and 0.4ng on column standard is desired or this standard may be used for the 0.5ng/L concentration as well) for SCAN and 0.2ng/L, 4.0ng/L, and 200ng/L for SIM.

Position 1 – Total Air Flow (Zero Air)
Position 2 – Standard Flow (Purchased Standard One)
Position 3 – Standard Flow (Purchased Standard Two if Applicable)
Position 4 – Total Air Flow (Zero Air) (utilized if preparing a two dilution standard)
Position 5 – Diluted Standard Flow (utilized if preparing a two dilution standard)

Step1: Determine the required flow rate of the stock standards (positions #2 and #3). The range must be from 5 to 50sccm (standard cubic centimeters per minute, same as ml/min). The flows listed below are guidelines to be used for the default standard flow (based on the desired standard concentration) and were chosen based on the ultimate final dilution required and limitations of the Dynamic Diluter (flows must be from 150 to 2000ml/min.).

Desired Standard Conc.	Default Standard Flow
200ng/L	50ml/min
100ng/L	50ml/min
20ng/L	20ml/min
5.0ng/L	10ml/min
4.0ng/L	8ml/min

1ng/L	50ml/min; 20ml/min (See Note 1 below)
0.2ng/L	10ml/min; 20ml/min (See Note 1 below)

Note 1: For the 1ng/L and 0.2ng/L standards (or any standard requiring more than a 400X dilution of the stock), a slightly different procedure is performed. In order to prepare these standards, a double dilution must be performed which involves taking the primary dilution flow and making a secondary dilution of that using the diluent gas. Unscrew the cover of the dilutor and connect the first mass flow controller as well as the tubing to re-route the first dilution output from the final standard Summa canister to the 2nd dilution chamber. Refer to example 2 for the calculation guidelines to prepare a two dilution standard.

Example 1: Prepare a 200ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required dilution factor for each stock.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 200ng/L = 5

Step 3: Calculate Total Flow
Total Flow= (stock std. flow-see table above)*(Dilution Factor)
Total Flow=50ml/min*5 = 250ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=250ml/min-(50+50)ml/min = 150ml/min

Example 2: Prepare a 0.2ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required total dilution factor for the 0.2ng/L standard.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 0.2ng/L = 5,000

The two dilutions must be performed which total the dilution factor calculated above. Since the flow for the Diluter is restricted to a maximum of 2000ml/min, the total flow (as calculated in Step 3 below) cannot exceed 2000ml/min; therefore, the dilutions must be chosen accordingly.

Step 3: Calculate Total Flow
Total Flow = (stock std. flow-see table above)*(Dilution Factor)
Total Flow (Dilution 1) = 10ml/min*200 = 2000ml/min

For the 2nd dilution take the stock standard flow selected for dilution 1 for the two purchased cylinders (10ml/min each based on the desired final concentration) and add them together (10ml/min + 10ml/min for 20ml/min) to get the stock standard flow for the 2nd dilution.

2nd Dilution Factor Needed = Total Dilution/1st Dilution
2nd Dilution Factor = 10000/200(1st dilution) = 50

Total Flow (Dilution 2) = 20ml/min*50 = 1000ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=2000ml/min-(10+10)ml/min = 1980ml/min (Dilution 1)
Air Flow=1000ml/min-20ml/min = 980ml/min (Dilution 2)

Position 1 = 1980ml/min
Position 2 = 10ml/min
Position 3 = 10ml/min
Position 4 = 980ml/min
Position 5 = 20ml/min

Step 5: Enter flow rates in the appropriate fields in the Entech software. Start flows by clicking the "GO" button in the top right of the window. Allow flows to equilibrate for at least fifteen minutes, then attach an empty canister to the outlet port and open the valve. The outlet pressure will be displayed in the lower right of the window, in units of psia. Close the canister valve when the pressure reaches 30psia. There is a relief valve on the diluter that will open when the pressure reaches 35psia, so the canister will still be usable if the valve is not closed in time.

10.2.2.2 When analysis of additional (extra) compounds are requested which are not in the purchased stock cylinders, the following preparation instructions should be used. In addition, the internal standard / surrogate standard may also be prepared in this manner (Sections 10.2.2.2.1 – 10.2.2.2.2) as mentioned in Section 10.2.1.

10.2.2.2.1 *Equi-mass "soup"* (contains compounds in equal mass amounts) or *cocktail* prepared from the neat compounds for a large number of components. If additional SIM compounds are requested, the same cocktail may be used.

Cocktail Preparation:

Step 1: This cocktail is prepared by combining 25mg of each neat compound into a small glass vial. Use a microliter syringe to transfer each compound, cleaning with solvents in between. Put the vial in the freezer between aliquots to minimize volatilization. Take the density of each compound into account to determine the actual amount of each compound to spike into the cocktail by using the following equation.

$$S = \frac{A}{D} \qquad \text{(Equation 4)}$$

Where:

S Actual spike amount (µL)
A Desired amount for each compound (mg)
D Density (mg/µL); refer to Table 2 for the density

Example: The actual volume of acrolein to add to the cocktail is calculated by the following.

$$S(Acrolein) = \frac{25mg}{\left(0.840\frac{mg}{\mu l}\right)} = 29.8\mu L$$

Step 2: The concentration of each compound in the cocktail is determined by the following equation.

$$C = \frac{A}{V}\left(1000\frac{\mu g}{mg}\right) \qquad \text{(Equation 5)}$$

Where:

C Concentration of cocktail (µg/µL)
A Amount of each compound (mg)
V Final volume of cocktail (total spike volumes of each compound) (µL)

Example:

$$C = \frac{25mg}{631.8\mu L}\left(1000\frac{\mu g}{mg}\right) = 39.569\mu g/\mu L$$

10.2.2.2.2 *An intermediate standard* is prepared from neat compounds by spiking individual compounds into a glass static dilution bottle (SDB) as described in Section 10.2.1.1 or spiking an aliquot of a cocktail into the SDB. The spike amount of a cocktail is determined by using the following equation.

$$S = \frac{C_1 V}{C_2} \qquad \text{(Equation 6)}$$

Where:

S Spike amount required in order to obtain the desired concentration (µL)
C_1 Desired concentration of SDB (µg/mL)
C_2 Concentration of cocktail (µg/µL)
V Volume of SDB (L)

Example: Determine the spike amount of the cocktail required to achieve the desired intermediate standard concentration.

$$S = \frac{\left(1\frac{\mu g}{ml}\right)(2010ml)}{27.81\frac{\mu g}{\mu L}} = 72.28\mu L$$

10.2.2.2.3 *Intermediate Standard Preparation (Gaseous Compounds*) As an alternative to the glass SDB method, if the extra compounds needed to be analyzed are gases at room temperature, use a gastight syringe to prepare an intermediate standard in a 1L Tedlar bag filled with humidified zero-grade air. Use the molecular weight of the compound to calculate the microliter amount to be spiked into the bag to achieve desired concentration. The spike amount is determined by using the following equation.

$$S = \frac{C * V * 24.46}{M * \left(1000\frac{ng}{\mu l}\right)}$$

S	Spike amount required in order to obtain the desired concentration (µl)
C	Desired concentration (ng/L)
V	Volume of the Tedlar Bag (1L)
M	Molecular Weight of the compound
24.46	Molar Volume of gas at 25°C, 1atm

Example:

Make a 100,000ng/L intermediate standard of Chloro-difluoromethane (Freon22) in a Tedlar Bag, where M=86

$$S = \frac{100{,}000\frac{ng}{L} * 1L * 24.46}{86 * \left(1000\frac{ng}{\mu l}\right)} = 28.44\mu l$$

10.2.2.2.4 *The Working standard* for extra compounds is prepared in a Summa canister by spiking an aliquot of the intermediate standard (glass SDB or Tedlar bag) using a heated gastight syringe. The preparation of these standards shall follow the instructions detailed in Section 10.2.1.2. The concentrations for working standards are usually 20 and 200ng/L, however different concentrations can be chosen which work best for a particular project.

10.2.3 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) Prepare a secondary source standard (either a different manufacturer or different lot from the same manufacturer as the initial calibration standard) using the same procedures as the primary source. The ICV/LCS working standard should

contain each target analyte present in the calibration working standard. Prepare the ICV/LCS working standard at a concentration of 200ng/L. Differing injection volumes account for the allowed concentrations listed in Table 4 for SCAN and 4A for SIM. The preparation of this standard shall follow the instructions detailed in Section 10.2.2, using the certified second-source standard cylinder.

10.2.4 Continuing Calibration Verification (CCV) Standard The CCV is the same as the initial calibration working standards detailed in Section 10.2.2.

10.2.5 Screening Standards Recommended procedure: Prepare a 0.5ug/mL and/or a 3.0ug/mL concentration standard so that the GC may be calibrated utilizing a few levels (may include approximately 0.5ng, 150ng and 600ng). However, other concentrations can be prepared depending on the desired range.

Any of the desired standard concentrations (primary and secondary) may change as long as the equations and the appropriate densities remain the same.

10.3 Storage and Expiration Dates

All standards that are to be stored in a freezer shall be stored at ≤-10˚C for DoD projects.

- Neat Stock Liquids are stored at < -10˚C (-10˚C to -20˚C) as specified by the manufacturer or for a period of five years.
- Equi-Mass Primary Stock Standard is a cocktail or soup of neat compounds (containing compounds in equal mass amounts) used to in preparing intermediate gas phase standards and shall be stored in the freezer at < -10˚C (-10˚C to -20˚C) for up to six months. This is assuming that the soup is sealed with a septum-containing screw cap or Mininert™ valve. The selection of the compounds for the soup should be performed in accordance with the guidelines in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Purchased Stock Standards Cylinders must be stored at laboratory temperature for a period of 2 years or as specified by the manufacturer before vendor re-certification or purchase of new standards.
- Intermediate Calibration Standards prepared by static dilution must be stored in an oven at a temperature of approximately 60°C to ensure analyte vaporization. Every time a standard is prepared from the static dilution bottle (SDB), the concentration changes. To increase the useful lifetime of an SDB standard, remove volumes of 25mL or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, an SDB intermediate standard is stable for approximately two months as long as new working standards made from this standard continue to meet acceptance criteria. These bottles must be in the oven for a minimum of one hour prior to use in preparing working standards. The guidelines for the storage and expiration date for the intermediate calibration standards are stated in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Prepared Stock / Intermediate Calibration Standards prepared in Summa canisters (1000ng/L) may be stored at laboratory conditions for up to three months in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

- Calibration or Working Calibration Standards prepared in canisters may be stored at laboratory conditions for one month in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

11) Method Calibration

11.1 Initial Calibration

The initial calibration is performed to determine instrument sensitivity and the linearity of the GC/MS response for the target compounds.

Initial calibration requirements are as follows:

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. An initial calibration must be performed at a minimum initially per instrument, annually thereafter or whenever the continuing calibration verification standard does not meet the acceptance criteria.
3. Highest concentration, together with the lowest concentration, defines the calibration range.
4. The method reporting limit for any reported analyte must be at >/= the lowest calibration point.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from lowest to highest concentration.
8. All ICAL analyses must be completed within the 24-hour tune window.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. One of the calibration points from the initial calibration curve must be at the same concentration as the continuing calibration verification standard.
11. The upper end of the calibration range must not exhibit any peak saturation for any analyte or the range must be lowered accordingly.
12. The initial calibration model must be linear calibration using average of response factors and cannot be changed for any reason.
13. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at or below the MRL (LOQ) and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
    - Points at the high end may be dropped, but doing so lowers the calibration range.
    - Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).
    - Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but

the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.

- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 24 hours).
- Once the ICAL has been used to calculate and report sample results it MUST not to be changed for any reason.
- It is recommended that if an analyte has a higher MRL than the lowest concentration analyzed that the low standard be automatically dropped from the curve (i.e., acetone MRL is 5, drop at least the 0.4ng point).

11.1.1 Calibration Points Analyze the calibration standards (analyze low to high) that span the monitoring range of interest of the samples. For SCAN, the range is typically 0.4ng-100ng on column; however, 0.08ng on column may be added if low level analyses are requested. For SIM, the range is 10pg on column to 50,000pg on column. The dynamic range is dependent on the sensitivity of a particular instrument as well as the required reporting limit for a given project and may be adjusted accordingly. Refer to Table 3 (SCAN) and Table 3A (SIM) for the concentrations of the compounds of interest in the initial calibration at each particular calibration concentration level.

Note: Refer to the EXCEL TO-15 Standard Concentration templates, located on the network at Q:\\TO15 Std. Concentrations\Std. Conc. Templates for both the SIM and SCAN templates. These templates must be utilized for the documentation of the standard canister concentration selection, final ICAL level concentrations and the determination of the correct injection volumes for the selected standard canister concentrations. If the primary or secondary stock standard cylinder concentrations are revised (upon re-certification or new purchases), the EXCEL spreadsheet templates, injection amounts and the ICAL concentrations in each instrument method must be adjusted accordingly. Other templates may be employed as long as they are validated and provide at least the same information.

SCAN

1. Determine if the lower end of the calibration range is to be 0.08ng or 0.4ng on column. If the low end is 0.08ng, then the 1ng/L standard must be utilized.
2. Determine if the 1ng/L or 20ng/L standard canister is to be used for the 0.4ng on column point.
3. Follow the instructions in the spreadsheet and save the file under the correct instrument folder and the initial calibration method identification.
4. Print the final ICAL concentration sheets and place into the corresponding ICAL folder

11.1.2 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not met and at least annually. The following procedure must be followed when updating an initial calibration method.

1. Open the most recent method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID must be the date files were analyzed.

3. Quantitate midpoint standard and check retention times and integrations. Update retention times if necessary using QEdit or Easy ID (Tools → Easy ID). Requant if any changes are made and verify all peaks are identified correctly. Print.
   a. While midpoint standard is loaded update reference spectra (Continuing Calibration → Update Reference Spectra).
   b. With midpoint standard loaded update qualifier ion ratios and retention times (Initial Calibration → Update Levels → Select Update Level and then select Retention Times (Replace) and Replace Qualifier Ion Relative Responses).
   c. If necessary adjust integration parameters prior to processing remaining ICAL points.
4. Quantitate remaining ICAL standards. Review each peak for retention time, integration, and print. Review low level standards for acceptable signal to noise ratios and high level standards for saturation.
5. All responses must be cleared from ICAL before updating (Initial Calibration → Clear All Calibration Responses).
6. Update responses for each standard level (Initial Calibration → Update Levels) or (Initial Calibration → Quick Levels Update). If Quick Levels Update is used do not requant datafiles.
7. Save method.
8. Check Response Factor Report and evaluate whether any points should be dropped following the criteria outlined in this SOP.
9. Save method if any changes are made.
10. Verify calibration files listed on Response Factor Report are correct.
11. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report.

11.1.3 Analytical Window If time remains in the tune window after meeting the acceptance criteria for the initial calibration, samples may be analyzed according to the procedure described in this document (see Section 12.3.2). If time does not remain in the analytical window, a new sequence shall commence with the analysis of the instrument performance check compound (BFB) and the continuing calibration verification standard.

11.1.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 12.4. Use the standard prepared in accordance with Section 10.2.2 of this SOP. Attach the calibration standard and internal standard/surrogate canisters to the designated inlets on the preconcentrator and open the canister valves. Analyzing different volume aliquots of the calibration standards produces differing concentrations.

Analyte responses (target ion areas) are tabulated and recorded using the Enviroquant program. Quantitation ions for the target compounds are shown in Table 2 and 2A and the primary ion should be used unless interferences are present, in which case the secondary ion may be used, but the reason documented in the initial calibration file and all subsequent quantitations utilizing that ICAL must be performed using the same ion selections. Refer to Section 15.2 for the required calculations and Section 16.4 for the acceptance criteria.

11.1.4.1 Additional Requirements The procedure for performing and generating a new initial calibration method must follow a few additional requirements.

1. If any analyte lacks the appropriate sensitivity (3 to 1 signal to noise ratio) at the low end of the calibration range, this point must be dropped from the curve and the MRL/LOQ raised accordingly.
2. No detector saturation may occur for any compound; the upper calibration level must produce no saturated peaks. Exhibited by:
   - The flattening of the response for the higher concentration standards as shown on the plot;
   - The presence of a reverse tail or rise on the front part of the peak;
   - The observed actual percent ratio of the secondary ion presence is lower than the expected percent ratio; or
   - The presence of a flat topped peak and again by the decline or saturation of the secondary ion compared with the expected % recovery.

11.1.4.2 LOQ Establishment, Verification and Acceptance Criteria

1. The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
2. The LOQ for each analyte must be ≥ the analyte's LOD.
3. Initially a passing demonstration of precision and bias must be performed at the LOQ.
4. Run CCV 2 times at LOQ and:
   a. Generate a duplicate report for precision using ±25% as the criteria.
   b. Check the %Rec using laboratory generated control limits.
   c. Check the signal to noise ratio (S/N) using the software. The S/N ratio must be at least 3:1 for each analyte.
   d. All ion abundances must be acceptable per the requirements set forth in this document.
5. If any compounds fail, verify at a higher level and notify reporting. Also, make a note in the ICAL documentation.
6. Turn in all LOQ verification data (quant reports and software reports/checks) to QA (regardless of pass/fail).
7. Verify the LOQ on each instrument quarterly.

11.1.5 Initial Calibration Review Analyst's calculation and assessment along with a peer review of all ICAL data and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples. In the case where samples are placed on the autosampler and allowed to run overnight, the sample results may only be reported if the ICAL is reviewed and found to be acceptable. The ICAL checklist in Attachment 2 must be used to document the review and approval process.

Perform a review of specific aspects of the calibration which might compromise data quality such as inappropriate extension of the calibration range with detector saturation and/or a lack of sensitivity for any analyte. Analyte concentrations which do not meet the signal to noise ratio or exhibit saturation are not to be reported and must be eliminated from the initial calibration. These instances should be followed by a short explanation regarding the reason for the omission.

11.1.6 Initial Calibration File An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- BFB tune analysis report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and % recovery report.
- TO-15 Standard Concentration Spreadsheet (exact ICAL level concentrations and ICV concentrations)
- Any manual integration documentation

11.2 Initial Calibration Verification Standard

Verify the initial calibration by analyzing an initial calibration verification standard (ICV). This standard shall be obtained or prepared from materials acquired from a different manufacturer or lot from that of the initial calibration and prepared according to Section 10.2.3.

Analyze 50ng or less (refer to Table 4 for the secondary source standard concentrations) of the ICV standard depending on the dynamic range of a given instrument and refer to Section 15.4 for the required calculations.

12) Sample Preparation/Analysis

12.1 Sample Preparation

The pressure/vacuum is checked and the canister pressurized upon receipt by the laboratory, as needed. When necessary, canisters shall be pressurized with humidified zero grade air. However, if the samples are to be analyzed in accordance with EPA Method 3C then the samples must be pressurized with UHP Helium (refer to Section 12.9 for additional information). The client must be made aware of this in advance and given the option of either submitting two canisters for analysis or receiving a report with qualified results (TO-15 Modified).

Depending on the size of the canister and location of sampling and as specified in the SOP below, samples may be pressurized to approximately 1.0psig to 3.5psig. Additional information may be found in the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters*. Initial and final pressures are recorded in LIMS and should be repeated on the back of the sample tag. The dilution factor created by filling the sample canister is calculated using equation number 12 in Section 15.7.

12.2 Screening

The analyst must screen a sample or subset of samples if the source is of unknown origin. Typically, if the source is known to be indoor or ambient outdoor air, no screening is necessary. However, if screening is required make sure that the instrument is calibrated. A single point calibration is sufficient; however, the instrument may be calibrated utilizing a two point calibration. The ICAL points are recommended to be at approximately 0.5ng, 150ng and/or 600ng spanning the desired dynamic range. Refer to Section 10.2.5 for additional information.

Inject a 1mL or smaller aliquot of each sample into a GC/flame ionization detector (FID) system that has been calibrated with a standard containing a subset of the target analytes. This subset represents the most commonly found compounds in air samples, such as acetone, trichloroethylene, and toluene. Use the results to determine the maximum volume of sample to be analyzed by TO-15 by utilizing the following equation. Dilutions may be prepared as necessary according to Section 12.9.1.

$$I = \frac{C}{H}$$

Where:

I Injection volume (mL)
C Maximum calibration level (ng on column)
H Compound screening concentration (ng/mL)

Example: Select the compound with the highest concentration (toluene = 1.0ng/mL). If the upper calibration level is 100ng on column, then the following calculation determines the maximum injection volume to analyze.

$$\frac{100ng}{1.0ng/mL} = 100mL \text{ maximum injection volume}$$

12.3 Analytical Sequence and Data System Setup

12.3.1 Data System For the Tekmar AUTOCAN, fill in the sequence log of the Teklink program with the appropriate information. Refer to the Section 12.4.1 for the operating parameters.

For HP Chemstation, load the appropriate acquisition method for the GC/MS in the top window of the Chemstation program. Suggested GC/MS operating parameters are given in Section 12.4.2.

12.3.2 Analytical Sequence The analytical sequence must be completed for the analysis of ≤20 (19 samples including dilutions with one laboratory duplicate) field samples. A method blank (MB) shall be run to monitor for laboratory introduced contamination. There must be at a minimum a laboratory duplicate (LD) analyzed in each batch to access batch precision. The following generalized analytical sequence is to be followed:

Analytical Sequence Guideline

With Calibration	Tune Check[1] Calibration Standards (5 Standards Minimum) ICV Standard[2] (Acts as the ICV and LCS) QC Canister Checks[6] MB[7] Sample(s) – 1-19 Laboratory Duplicate[4]
With Continuing	Tune Check[1] CCV Standard[5] QC Canister Checks[6] MB[7] LCS[3] MRL Check Standard[8] Sample(s) – 1-19 Laboratory Duplicate[4]

[1] The instrument performance check solution must be analyzed initially and once per 24 hour (or as specified by the project) time period (sequence / tune window) of operation. All analyses for a sequence must be initiated (injected) prior to the expiration of the tune window.

[2] In this scenario, the ICV may also be evaluated as the LCS (differing acceptance criteria).

[3] An LCS shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard analyzed at the lower end of the calibration curve (below the midpoint).

[4] A laboratory duplicate must be analyzed at a rate of 1 per 20 or fewer samples. The duplicate must be rotated among clients, whenever possible. Also, a duplicate laboratory control sample may be analyzed to assess precision to meet project requirements or due to sample matrix effects.

[5] A CCV must be analyzed at the beginning of every analytical sequence.

[6] Any number of QC check canisters may be analyzed in the sequence to determine a canister cleaning batch or batches acceptability.

[7] Any of the QC Check Canisters may serve as the method blank as long as the minimum requirements detailed in this document are met. A method blank shall be analyzed at a rate of 1 in 20 or fewer samples.

[8] A MRL check standard may be analyzed with each batch of 20 or fewer samples (when an initial calibration is not analyzed within the same batch). Additional information is included in Section 12.15.

Note: Client project batch specifications may require certain modifications to the analytical sequence; however, a batch may not be more lenient than that which is specified in this document.

12.4 Conditions

12.4.1 Sample Collection Conditions The suggested settings and system parameters are as follows:

Adsorbent Trap

Set Point:	35˚
Sample Volume:	up to 1L
Dry Purge:	300mL
Sampling Rate:	100mL/min (utilize for a sample injection volume of >100mL); 40mL/min (utilize for a sample injection volume of 25-100mL)
Desorb Temp.:	200˚C to 230˚C
Desorb Flow Rate:	8-10mL/min He
Desorb Time:	3.0 minutes

Refocusing Trap

Temperature:	-180˚C
Injection Temp.:	160˚C
Injection Time:	1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature:	265°C

Initial Bakeout: 2 hours or until clean blank is obtained
After each run: 5-8 minutes

Sample Run Time

Each analytical run is approximately 20 minutes long; the total cycle time is about 30 minutes between injections.

12.4.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

Item	Condition
Carrier Gas	Helium
Flow Rate	1.0-1.6mL/minute
Temperature Program	Initial Temperature: ~20˚C Initial Hold Temperature: 3 minutes Ramp Rate: 5˚C/min to 80˚C 2nd Ramp: 10˚C/min to 160˚C 3rd Ramp: 20˚C/min to 240˚C for 5 min hold
Detector B (MSD *Interface)*	260˚C
Electron Energy	70 Volts (nominal)
Mass Range (Scan mode)	34 to 280 amu
Mass Range (SIM mode)	Scan masses corresponding to the target analytes
Scan Time	To give at least 10 scans per peak, not to exceed 1 second per scan.

Note: The instrument may be operated in Selective Ion Monitoring (SIM) mode if requested by the client.

12.5 Instrument Performance Check

Since the BFB tuning compound is included in the internal standard and surrogate standard canister and an autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

The mass spectrum of BFB must be acquired in the following manner.

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.
- All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.
- The ion abundance criteria must not be changed from the requirement stated in this document (TO-15 or TO-14A, as requested).

All subsequent standards, samples and QC samples associated with a BFB analysis must use identical instrument conditions.

12.6 Continuing Calibration Verification Standard

Verify the calibration each working day, where necessary (e.g., an ICAL was not analyzed or the tune window has closed) by analyzing a continuing calibration verification (CCV) standard from the initial calibration standard canister. The concentration of the calibration verification may be varied between the low calibration standard and the midpoint of the calibration range; however, the concentration must be at one of the levels analyzed in the initial calibration. Refer to Table 3 for the standard concentrations. Refer to Section 15.3 for the required calculations.

12.7 Canister Quality Control Check and Method Blank

The method blank must be a sample of a matrix similar to the batch of associated samples that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure, and in which no target or interferences are present at concentrations that impact the analytical results for sample analyses. Prepare a canister that has not left the building by pressuring with humidified zero air. Analyze an aliquot of one liter along with the same volume of internal standard and surrogate as standards and samples. Additionally, a blank must be analyzed whenever a high concentration sample is encountered and carryover is suspected.

A Quality Control (QC) check canister pressurized with humidified zero air may serve as a method blank as long as the analyte concentration requirements stated in the canister quality control check section (Sections 16.7 and 16.8) and other requirements (refer to Section 16.12 for internal standard requirements) are met. Assuming continuing failure, another QC canister or a new canister must be prepared and analyzed in order to verify that no system contamination exists. For tracking purposes the unique laboratory barcode given to a canister shall be the information included in the sample analysis identification.

12.7.1 Sampling Systems Section 7.1 and 8.4 of Method TO-15 describe the setup and certification procedure for a specific sampling apparatus that has been used by the EPA for several of its large air monitoring programs. These systems are rarely used for the types of projects that make up the bulk of the laboratory's work. The vast majority of samples analyzed by the laboratory are taken into Summa canisters either as grab samples or using a simple time integrated sampling device (flow controller), as in Section 8.2.1 of the method, so these procedures are not part of the typical protocol for providing sampling materials to clients. The laboratory has developed an SOP for the cleaning and certification of the materials it provides its clients for obtaining air samples to be analyzed by method TO-15. Refer to the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters* for additional information.

It is this laboratory's interpretation that the sampler system certification procedure described in Section 8.4.4 of the TO-15 method applies to the specific sampling apparatus described in the method and not to the sampling procedures used by our clients. The laboratory does not maintain a dynamic calibration manifold or canister sampler apparatus as described in the method and thus performance of the relative accuracy certification procedure described in section 8.4.4 is not possible.

12.8 Laboratory Control Sample

The laboratory control sample is a sample matrix, which is free from the analytes of interest and spiked with a standard containing known amounts of analytes. The

laboratory control sample is an injection of the initial calibration verification standard. Inject the LCS (ICV) at concentrations below the midpoint of the calibration curve. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

12.9 Sample Analysis

Prior to analysis, all sample containers (canisters and bags) should be at temperature equilibrium with the laboratory.

- Attach sample canisters to Tekmar AUTOCan using a 9/16" wrench. Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments). Tedlar bags can be connected using soft silicone tubing or a 3/16" fitting with a reusable ferrule.
- Before opening the valve, check for leaking fittings by running the leak check program in the Teklink software. Quick connect fittings must be leak checked before connecting the sample container.
- If system is leak tight, open the canister valves and start the automated preconcentration procedure. Make sure the Chemstation data acquisition software has been readied.
- Maintain the trap at an elevated temperature until the beginning of the next analysis.

Check all target compounds using the QEdit routine in Enviroquant, making sure all extracted ion chromatogram peaks are integrated properly (see Section 12.13).

Note 1: The secondary ion quantitation is only allowed if there is sample matrix interference with the primary ion. If the secondary ion quantitation is performed, document the reasons in the instrument run logbook and/or on the quantitation report (initial and date any notation).

Note 2: Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fitting higher than 80ºC.

SCAN Mode - The instrument is normally operated in the SCAN mode, where the following procedure may be followed.

- Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic range from 34 to 270 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning allows identification of unknown compounds in the sample through searching of library spectra. See operating conditions in Section 12.4.
- Generate a quantitation report for each run.
- If reporting Tentatively Identified Compounds (TICs), refer to Section 12.9.2 for identification criteria.

SIM Mode - When the client requests SIM mode, select SIM instead of SCAN mode and identify a minimum of two ions per analyte of interest. Also, a minimum of two ions for each internal standard and surrogate compound should be selected.

Helium Pressurization – If a canister is pressurized with helium, a correction factor is applied to sample volumes extracted from the canister via auto sampler. This is due to the difference in thermal properties between helium and air. A correction factor worksheet has been generated to determine the exact volume taken from a canister and may be found at J:\\A-GCMS\Helium Pressurization. Save file, print the sheet and include with the data. Refer to the instruction page in the template for all of the instructions and calculations including backfilled canisters.

AutoCAN Leak Checks – Canisters should be put on at least two different AutoCAN positions to confirm a "leak". In addition, the valve threads should be inspected for defects which may prevent a good seal with the AutoCAN. Once a canister has "failed" the leak check it must be tagged, an NCAR initiated, and the PM notified. Regardless of what the client or PM specifies as the fate of the sample, the canister must be put on maintenance hold to complete a full 24-hour leak check. A yellow sheet is to be completed in addition to, but not in lieu of an NCAR. This is a fixed QA procedure with no allowance for deviation.

12.9.1 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot should be analyzed. The dynamic range of volume aliquots for the automatic cryogenic concentrator is 20cc to 1L. If a volume smaller than 20cc is to be analyzed, a dilution should be made in a Tedlar bag, or the sample directly injected using a gastight syringe. Guidance in performing dilutions and exceptions to this requirement are given below.

- Refer to Section 12.4.1 (Adsorbent Trap Sampling Rate) for the required sampling rate if less than 100mL is to be analyzed.
- Use results of the original analysis to determine the approximate dilution factor required and get the largest analyte peak within the initial calibration range.
- The dilution factor must be documented (and included in the final report) and chosen in such a way as to keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument.

Tedlar bag dilution:

- Make a dilution by filling a Tedlar bag with 1.0 liter of humidified zero air using a one-liter gas syringe.
- Calculate the volume of balance gas needed to obtain the required dilution.
- Remove the difference in the balance gas using a syringe.
- Add the calculated sample amount using a gastight syringe.

Direct injection:

- Make a direct injection by attaching a clean, humidified zero air filled Summa canister to the preconcentrator autosampler using 1/4" stainless steel or teflon tubing with a "tee" septum port. This canister should be the same canister that may be used as the method blank.
- Inject the sample through the septum while the preconcentrator withdraws a 200cc aliquot from the canister.

12.9.2 Tentatively Identified Compounds When requested, a mass spectral library search may be made for the purpose of tentatively identifying sample components not associated with the calibration standards. The necessity to perform this type of identification will be determined by the purpose of the

analyses being conducted. Data system mass spectral library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Certain programs may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. The following guidelines are used for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. For example, for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30 and 70%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- The concentration of the tentatively identified compound is estimated by assuming a response factor of 1.0 and comparing the response of the tentatively identified compound to the response of the nearest internal standard.
- If non-target analytes are not Q-deleted from the quant report, the analyst must evaluate whether these compounds should be reported as TICS.

12.10 Duplicate

A duplicate must be analyzed to assess laboratory precision and samples selected for duplicate analysis shall be rotated among client samples, where applicable. Some projects or sample matrix issues may require the analysis of a duplicate laboratory control sample (DLCS).

12.11 Internal Standard (IS)

The concentration of internal standard added to each standard, field sample and QC sample must be consistent from that of each current ICAL standard.

12.12 Surrogates

Internal standards/surrogates must be added at the same volume for every standard, sample and QC sample. Surrogate compound recoveries are requested by a number of clients, but are more appropriately used as system monitoring compounds. This is due to the fact that the compounds are introduced directly into the analytical system and not into the canisters or bags. It is for this reason that they are not considered to be true surrogates and a fixed window is applied. Additionally, surrogates are not included in the ICAL because they are not required by the method and are only system monitoring compounds.

12.13 Manual Integration and Q Deletion

A list of abbreviations (codes) that may be used to give a reason for performing either of these procedures are listed in the *SOP for Data Review and Reporting*.

12.13.1 Manual Integration The integration for each peak must be legally defensible and shall be checked to ensure that it has been integrated properly and consistently between samples, standards and QC samples. All peak reviews and manual integrations must follow the requirements specified in the *SOP for Manual Integration Policy* and the *SOP for Laboratory Ethics and Data Integrity.* The requirements in the above stated procedure include when manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., chromatograms showing both the integration prior to any manual integrations and those depicting the corresponding manually integrated peaks), and notation of rationale, date, and initials of person performing the manual integration operation. In addition, manual integrations must be reviewed and approved by a second reviewer and the manual integrations maintained in the appropriate job file.

Reporting Requirements Certain project requirements including samples which are submitted under the Department of Defense (DoD) QSM require that the case narrative include an identification of samples and analytes for which manual integration is required. Refer to project requirements to determine if this is necessary.

12.13.2 Q Deletion Q deleting may be performed to either delete a false positive or delete non-target compounds.

12.14 Detection Limits and Limits of Detection

The MDL study shall be performed annually for all target analytes on each instrument (with identical configurations) for which this method is performed. The MDL shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The detection limit shall be used to determine the LOD for each analyte.

Once determined on each instrument, the highest LOD (for each analyte from all instrument determinations) shall be used as the uniform LOD. However, if a lower detection limit is reported, then the samples must have been run on that specific instrument on which the lower LOD was determined.

12.14.1 Performance and Acceptance Criteria

1. The MDL must be <0.5ppbV for each analyte (Method 11.11.1).
2. Perform Limit of Detection (LOD) verification on all instruments (performing this method) immediately following the MDL study. Spike the LOD at 2-4x the MDL; the spike level establishes the LOD.
3. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).
   - It is specific to each combination of analyte, matrix, method and instrument configuration.
   - The LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.
4. If the LOD verification fails (per #3), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.
5. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

12.15 Method Reporting Limit Check Standard

It is recommended to analyze a MRL check standard at the current MRL or required MRL for the batch (per client requirements) of twenty or fewer samples if the CCV fails low for any target compound. A MRL check standard may also be required per client specifications.

This check standard can also serve as the LOQ verification if it meets the specific requirements listed in Section 11.1.4.2. Apply the requirements and retain all documentation accordingly. Refer to Attachment 4 for Minnesota specified MRL check standard criteria.

12.16 Method Modifications

Method modifications are not allowed under NELAC\TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 12.9.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. There are multiple quantitation methods, which are subsets of the compound list in Table 2. Therefore, files will be named with an eight-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files are saved in a unique sub-directory on the server.

14.2 Sufficient raw data records must be retained on file of all laboratory analyses described in this document including passing QC canister checks, tune checks, instrument calibrations, verifications, sample analyses and dilutions, QC checks, and method detection limit studies. The information that is required includes: analysis/calibration date and time, test method, instrument, sample identification, analyte identification, analyst's initials, concentrations and responses, as well as standards used for the analysis and calibrations, all manual calculations including sample dilutions and manual integrations to permit reconstruction of analyses. Information entered and reported on the quantitation report and instrument run log must be complete and accurate. All data shall be obtained following defensible and ethical practices in accordance with the most recent Quality Assurance Manual and the *SOP for Laboratory Ethics and Data Integrity*.

Note: All data records must explicitly connect data to the initial instrument calibration. This includes all samples, continuing calibrations and QC samples.

14.3 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time (if the holding time is 72 hours) of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings and balance gas if pressurized with helium), standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 This method has specific requirements including the use of canisters; any modification must be reported accordingly. All reports that fall under the laboratory's certificate of approval (in accordance with NELAC/TNI standards) must include a statement(s) clarifying any deviations from the scope of this certification. Refer to Section 15.10 for additional information and specific items, which require this clarification.

15.2 Initial Calibration

Tabulate each of the following:

15.2.1 Equation Number 1 - **Relative Response Factor (RRF):**

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$ where:

A_x is the area response of the analyte quantitation ion.
A_{is} is the area response of the corresponding internal standard quantitation ion.
C_{is} Internal standard concentration, ng.
C_x Analyte concentration, ng.

Note: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC samples is the same from run to run.

15.2.2 Equation Number 2 - **Average (or Mean) RRF:**

$$\overline{RRF} = \frac{\sum_{i=1}^{N} RRF_i}{N}$$ where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

N is the number of calibration concentration levels.

15.2.3 Equation Number 3 - **Standard Deviation, SD:**

SD = $\sqrt{\sum_{i=1}^{N} \frac{\left(RRF_i - \overline{RRF}\right)^2}{N-1}}$ where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

$\overline{RRF}$ Average (or Mean) RRF of all concentration levels in the initial calibration curve.

N total number of calibration concentration levels

15.2.4 Equation Number 4 - **Percent Relative Standard Deviation, %RSD:**

%RSD = $\frac{SD}{\overline{RRF}}(100)$ where:

SD Standard Deviation calculated in equation number 3

$\overline{RRF}$ Average or Mean RRF

15.2.5 Equation Number 5 - **Relative Retention Time (RRT):**

$$\mathrm{RRT} = \frac{\mathrm{RT_C}}{\mathrm{RT_{is}}}$$ where:

RT_C Retention time of the target compound, seconds.

RT_{is} Retention time of the internal standard, seconds.

15.2.6 Equation Number 6 - **Mean Relative Retention Time ($\overline{RRT}$):**

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT_i}{n}$$ where:

$\overline{RRT}$ Mean relative retention time (seconds) for the target compound for all initial calibration levels.

RRT_i Relative retention time for the target compound in level i.

n Number of calibration levels

15.2.7 Equation Number 7 - **Mean Area Response ($\overline{Y}$):**

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$ where:

Y_i Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

n number of calibration concentration levels

15.2.8 Equation Number 8 - **Mean Retention Times ($\overline{RT}$):**

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$ where:

$\overline{RT}$ Mean retention time, seconds
RT_i Retention time for the internal standard for each initial calibration standard, seconds.
n number of initial calibration levels

15.3 Continuing Calibration Verification

- Calculate the (RRF) of each target compound using equation number 1.

15.3.1 Equation Number 9 - **Percent Difference, %D:**

$$\%D = \frac{RRFx - \overline{RRF}}{\overline{RRF}}(100)$$ where, for any given analyte:

RRF_x is the RRF from the CCV being evaluated.
$\overline{RRF}$ is the mean RRF from the current calibration curve.

15.4 Percent Recovery – ICV, LCS, Surrogates, MRL Check Standard

15.4.1 Equation Number 10 - **Percent Recovery (%R):**

%R = X/TV x 100

where
X = Concentration of the analyte recovered
TV = True value of amount spiked

15.5 Duplicate Analysis

15.5.1 Equation Number 11 - **Relative Percent Difference (RPD):**

$$\frac{x_1 - x_2}{\overline{x}}(100)$$ where:

x_1 First measurement value
x_2 Second measurement value
$\overline{x}$ Average of the two values

15.6 Internal Standards (IS)

- Calculate the mean area response $\overline{Y}$ for each internal standard using equation number 7.
- Calculate the mean of the retention times for each internal standard using equation number 8.

15.7 Pressure Dilution Factor (PDF)

15.7.1 Equation Number 12 - **PDF**, for samples collected in Summa canisters:

$$PDF = \frac{P_{atm} + P_f}{P_{atm} + P_i}$$ where:

P_{atm} is the ambient atmospheric pressure, 14.7 psi at sea level.
P_f is the final sample canister pressure, in psig.
P_i is the initial sample canister pressure, in psig. This will most often be a negative value (sub-ambient initial pressure).

15.8 Results

If a canister has been pressurized with Helium and the Tekmar AutoCan was utilized, refer to Section 12.9.

15.8.1 Equation Number 13 - For calculating analyte concentrations in a sample, the starting point is the nanogram amount generated by the HP Enviroquant software, which appears on the quantitation report.

$$ng_x = \frac{A_x ng_{is}}{A_{is}\overline{RRF}}$$ where:

ng_x is the nanogram amount of analyte *x*.
A_x is the area response of the analyte's quantitation ion.
A_{is} is the area response of the corresponding internal standard's quantitation ion.
ng_{is} is the internal standard amount, in nanograms.
$\overline{RRF}$ is the average or mean RRFs

15.8.2 Equation Number 14 - The final analyte concentration, C_x, in units of micrograms per cubic meter (µg/m³), is then calculated from the following:

$$C_x = \left(\frac{ng_x PDF}{V}\right)\left(\frac{1\mu g}{1000ng}\right)\left(\frac{1000l}{1m^3}\right)$$ where:

V is the sample volume analyzed, in liters.
PDF is the sample canister pressure dilution factor.

15.8.3 Equation Number 15 - To convert to units of parts per billion volume (ppbv):

$$ppbv = \frac{\mu g/m^3}{MW} x 24.46 \qquad \mu g/m^3 = \frac{ppbv}{24.46} xMW$$ where:

MW is the molecular weight (Table 2) of the analyte, in g/mole.
24.46 is the molar volume of an ideal gas at 298 K (25 °C) and 760 mmHg (1 atm), in liters per mole (l/mol).
C_x the final analyte concentration in micrograms per cubic meter.

15.8.4 Equation Number 16 – Helium Pressurization (Injection Amount)

Applicable to canisters pressurized with helium and injected utilizing the mass flow controller of the AutoCAN. For full instructions and calculations, refer to the 1st tab of the template located at: J:\A-GCMS\Helium Pressurization\MFC_GCF _backfill.

15.9 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated by analytical sequence following the Daily QC review checklist (Attachment 3). The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The Sample Review checklist (Attachment 3) is used to document sample review per service request and once completed, initialed and dated must be filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file organized by instrument and date. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.6.

15.10 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and information required by this laboratory's policy, NELAC\TNI standards, DoD Manual (applicable version, see reference section), client projects, and the TO-15 method including modifications, observances, data qualifiers, and certification information.

If the project requires that results be reported below the MRL (LOQ), but above the LOD all of the requirements specified for normal reporting apply (3:1 S/N ratio and ion abundance). This is regardless of the fact that the results will be qualified as estimated.

15.10.1 Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, must be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. All data qualifiers and flags should follow those listed in the most recent Quality Assurance Manual or as defined in any client requirements.

This form is necessary as a means for documentation. This form, among other information, will be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

15.10.2 NELAC\TNI Requirements

The following items do not comply with NELAC\TNI standard requirements and must be reported accordingly. A statement, however worded, must be

included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval.

- Reporting any compound which is not included in the second source standard (ICV or LCS) does not meet NELAC requirements.
- In addition, a report that contains a compound not included on the NELAC certificate of approval must also include the statement listed above.

15.10.2.1 Modifications

Method modifications are also not allowed under NELAC\TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 12.9.

15.10.3 Surrogates

Only report surrogates at the request of the client. If any surrogate is out of control, all samples results (with surrogates requested) associated with the surrogate must be reported with the appropriate data qualifier.

15.10.4 DoD Requirements

Report results with the appropriate data qualifiers, if samples cannot be reanalyzed for any reason. In addition and at a minimum, the following situations are to be noted in the case narrative: manual integrations, CCV out of control, and results exceeding the calibration range.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.2 Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate. Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV or any batch QC.

16.3 Instrument Performance Check

16.3.1 Acceptance Criteria

Refer to Tables 1 and 1A for the required ion abundance criteria.

16.3.2 Corrective Action Perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria. An acceptable tune is required for sample results to be calculated and reported.

16.4 Initial Calibration

16.4.1 Acceptance Criteria Refer to the following acceptance criteria for the initial calibration.

- The RRT for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound.
- The calculated %RSD for the RRF for each compound in the calibration standard must be less than 30% with at most two exceptions up to a limit of 40% (this may not be true for all projects).
- For each Internal Standard the area response (Y) at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range.
- The retention time shift for each of the internal standards at each calibration level must be within 20s of the mean retention time over the initial calibration range for each internal standard.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable ICV.

16.4.2 Corrective Action Follow the initial calibration requirements detailed in Section 11.1 for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards.

If the initial calibration results are outside the established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed, if reanalysis of the samples is not possible, data associated with an unacceptable initial calibration shall be reported as estimated with the appropriate data qualifiers.

16.5 Initial Calibration Verification Standard (ICV)

16.5.1 Acceptance Criteria The percent recovery for each compound in the ICV must be between 70%-130% for all analytes except vinyl acetate, which must be within 50-150%. Exceptions to this allowance for the vinyl acetate recovery are project specific requirements and any DoD type project, which shall adhere to the 70-130% requirement for all target compounds.

16.5.2 Corrective Action If the initial calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. Perform another initial calibration or if reanalysis is not possible, data associated with an unacceptable ICAL/ICV shall be reported as estimated with the appropriate data qualifiers.

16.6 Continuing Calibration Verification (CCV)

16.6.1 Acceptance Criteria All compounds must be evaluated prior to rounding. The percent difference for each target analyte must be within plus or minus 30% of the initial calibration average RRFs.

16.6.2 Corrective Action If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed; however, refer to 16.5.1 below.

16.6.2.1 Method Reporting Limit Check Standard

If the MRL check standard is unacceptable for any compound (sensitivity; ratio or %D), reanalyze at the same or higher level within the same batch and report data with the CCV flag and case narrative notes accordingly.

16.6.3 *DOD REQUIREMENT:* If a CCV fails, the laboratory must immediately analyze two additional consecutive CCVs (immediately is defined as within one hour).

- Both of these CCVs must meet acceptance criteria in order for samples to be reported without reanalysis
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

16.7 Canister Quality Control Check

The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included. If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual – DoD QSM, etc.).

16.7.1 Scan Analyses A canister is considered "clean" for normal SCAN analyses if the analysis shows <0.2ppbv of any target analyte (analyte exceptions listed in table below). If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual - DoD QSM, etc.).

Low Level SCAN Analyses For those analytes with a MRL of 0.1ug/m3, the QC criteria of <MRL is acceptable; otherwise, <0.2ppbV is required (analyte exceptions listed in table below).

SIM Analyses Results <MRL will be acceptable as this complies with the <0.2ppbV method requirement.

ANALYTE EXCEPTION LIST					
Compounds	ppbV	On Column (ng)	Compounds	ppbV	On Column (ng)
Target Analytes	0.2	0.50	Acrylonitrile	0.2	0.43
Chloromethane	0.2	0.41	Acetone	1.5	3.5
1,3-Butadiene	0.2	0.44	Ethanol	1.9	3.5
Acetonitrile	0.2	0.33	Vinyl acetate	0.99	3.5
Acrolein	0.65	1.5	1-Butanol	0.23	0.70
Isopropanol	0.28	0.70	Carbon Disulfide	1.1	3.5
2-Butanone	1.2	3.5			

Document the status of the check in LIMS and return the canister to the canister conditioning room. Additionally, if the check was found to be acceptable, the quantitation report must be kept on file for future reference

16.7.2 Tentatively Identified Compounds (TIC) If the batch of canisters are to be used for tentatively identified compounds (TIC) analysis, any non-target peaks present in the QC check canister analysis must be evaluated and determined to be less than the TIC reporting limit (10% of the internal standard). The concentration is estimated by assuming a RRF of 1.0 and comparing the response of the TIC to the response of the nearest internal standard.

16.8 Method Blank

16.8.1 Acceptance Criteria

- The concentration of a targeted analyte in the blank cannot be at or above the MRL, AND be greater than 1/10 of the amount measured in any associated sample. For any project that requires reported results less than the MRL, all associated measurements found in the MB should result in a qualifier; however, project requirements may differ and must be followed. Refer to DoD requirements listed below.
- The method blank should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.
- For DoD samples, the method blank will be considered to be contaminated if:

  1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
  2. The concentration of any common laboratory contaminant (acetone, ethanol, carbon disulfide, and methylene chloride) in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
  3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

16.8.2 Corrective Action If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results

are acceptable or prepare a canister per Section 12.7. If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). In all cases, the corrective action (reprocessing or data qualifying codes) must be documented. However, the specific corrective action depends on the type of project the blank is utilized for; therefore, refer (below) to the reporting/reprocessing requirements.

DEPARTMENT OF DEFENSE (DoD) QSM PROJECT: Any sample associated with a blank that fails the criteria shall be reprocessed in the same or subsequent analytical batch, except when the sample analysis resulted in a non-detect. If reanalysis is not performed, the results shall be reported with appropriate data qualifier.

OTHER PROJECT TYPE: Appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 18.4.

16.9 Laboratory Control Sample (LCS)

16.9.1 Acceptance Criteria Round all results to the nearest whole number prior to determining if the acceptance criteria have been met. The percent recoveries must be within the laboratory-generated limits and are referenced in the electronic TO-15 Method Manual. However, Arizona requires the percent recovery for each compound in the LCS to be 70%-130% (to match the ICV requirement). Therefore, the ICV exception for vinyl acetate stated in Section 16.5 requires the percent recovery for AZ samples to be 50-150%.

Note: Client project requirements, AFCEE and DoD requirements shall take precedence over the AZ requirement for AZ samples. Meaning if a sample is collected for a DoD project in AZ, DoD requirements specified in this document and the project specific QAPP (if supplied) are to be followed.

DoD Requirement: In the absence of client specified LCS reporting criteria, the LCS control limits outlined in the DoD QSM 5.0 Appendix C tables shall be used when reporting data for DoD projects.

16.9.2 Corrective Action If the LCS criteria are not met, determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and if the problem is with the injection re-analyze the LCS. DoD considers the same analyte exceeding the LCS control limits two out of three consecutive LCS to be indicative of non-random behavior; therefore, this trend should be monitored and the appropriate corrective action taken when it occurs.

16.10 Sample Results

16.10.1 Acceptance Criteria

- Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.

- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria described in this document.
- All target analyte peaks must be within the initial calibration range, diluted or reported with the appropriate data qualifier.

16.10.2 Corrective Action

- If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis as needed.
- If the area for any internal standard changes by more than ±40 percent between the sample and the most recent calibration, check for possible matrix interferences and re-analyze at a greater dilution. If the requirement is still not met and matrix interference is not detected the GC/MS system must be inspected for malfunction and maintenance made where necessary.
- When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

  To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.11 Laboratory Duplicate

16.11.1 Acceptance Criteria The relative percent difference must fall within ±25%. This RPD criterion also applies to duplicate laboratory control samples (DLCS).

16.11.2 Corrective Action If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.12 Internal Standards

16.12.1 Acceptance Criteria The following acceptance criteria must be applied to each run (except the ICAL – see Section 16.4).

- The area response for each internal standard in the blank must be within ±40 percent of the area response for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).
- The retention time for each internal standard must be within ±0.33 minutes of the retention time for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

16.12.2 Corrective Action

- Internal Standard Responses If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If

the problem is corrected with the lower volume analysis, report those results.

- Internal Standard Retention Times If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis where required.

16.13 Surrogates

16.13.1 Acceptance Criteria Since the matrix precludes the use of true surrogates and there is no established method criterion, acceptable surrogate recoveries are based on a fixed window of 70 - 130%. This is the typical requirement from clients. Additionally, these limits are referenced in SW-846 for use as guidance in evaluating recoveries. These limits are sufficient for evaluating the effect indicated for the individual sample results.

16.13.2 Corrective Action Poor surrogate recovery should be followed by re-analyzing a smaller aliquot to mitigate any matrix interferences. Evaluate the out of control surrogate for the effect on individual sample results.

16.14 Method Reporting Limit Check Standard

16.14.1 Acceptance Criteria Per client requirements or if the CCV is biased low for any compound, then evaluate the MRL check standard. Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio). Also, a percent difference +/-50% is recommended.

16.15 Sample Holding Time Expired

The customer is to be notified that the sample's holding time was missed and the customer is to decide if the sample analysis is to continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual for the appropriate data qualifier.

18.2 Initial Calibration and/or Initial Calibration Verification

All results reported with an unacceptable ICAL must be reported as estimated and all data shall be reported using defined qualifiers or flags or explained in the case narrative accordingly.

18.3 Continuing Calibration Verification

All results associated with an unacceptable CCV (other than #1 below) must be reported with the appropriate data qualifier, flag and/or explained in the case narrative.

1. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
2. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples with detects, then those detects must be reported with a qualifier, flag and/or explained in the case narrative.
3. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects must be reported with qualifiers, flags and/or explained in the case narrative as having less certainty. However, along with the data qualifiers, the case narrative may include information stating the fact that the results were not significantly affected if:
   a. *An MRL check standard was analyzed and found to be acceptable. The MRL must be the same as that analyzed in the MRL check standard for those analytes that were biased low in the CCV. Adjust MRLs (if required), flag data and state the certainty in the case narrative where the sensitivity of the instrument was demonstrated at the MRL; therefore, results were not significantly affected.*
   b. *With the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative and note procedure in case narrative.*
4. If the acceptance criteria was exceeded (biased high) for the CCV and there were detectable results in a sample, the results may be "qualified" if the results exceeded the regulatory/decision limit (this is to be stated in the case narrative along with the data qualifiers or flags).

18.4 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

18.5 Laboratory Control Sample

All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

18.6 Surrogate

Report sample results with the appropriate data qualifier.

18.7 Laboratory Duplicate

All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

18.8 Internal Standard

All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

18.9 Estimated Sample Results

18.9.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this SOP.

18.9.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

18.9.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty by reporting with the appropriate data qualifier.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

19.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 and 2A for both SCAN and SIM modes and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

19.3 Accuracy and Precision

Refer to Section 11.4 in the referenced method for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the electronic TO-15 Method Manual. Refer to Section 11.1.4.2 for the accuracy and precision requirements for concentrations at the LOQ/MRL.

19.4 Selectivity

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample

injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 98 or newer version
- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

19.5 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Therefore, ongoing demonstration of capable shall be performed and documented; however, the initial demonstration of method capability is not required.

19.6 Proficiency Testing (PT) Program

The laboratory shall participate in an air and emissions PT study for TO-15. The testing shall be performed in accordance with this document and meet the frequency and proficiency requirements detailed in the DoD QSM Version 5.0.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
22.0	03/21/15	C. Humphrey	Section 1 – Removed Note 1
			Section 4 – Revised section to include Hazard Assessment table
			Section 12.9 – Added Note 2
			Table 2A – Updated
			Table 3 – Updated
			Table 3A – Updated
			Table 4 – Updated
			Table 4A – Updated
			Attachment 3 – Added MAPH to Daily QC and Sample Review Checklists

21) References and Related Documents

21.1 EPA Method TO-14A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.2 EPA Method TO-15, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.3 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, January 1999.

21.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Addendum, January 17, 2002.

21.5 National Environmental Laboratory Accreditation Conference, *2003 NELAC Standard*, June 5, 2003, EPA 600/R-04/003 and 2009 TNI Standards.

21.6 *Preparation of Gas Phase Standards for Ambient Air Analysis,* Tekmar-DOHRMANN Application Note, Spring 96, Vol. 6.5.

21.7 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.

21.8 Arizona Administrative Code, Title 9. Health Services, Chapter 14. Department of Health Services Laboratories, December 31, 2006.

21.9 Florida Department of Environmental Protection, Chapter 62-160.

21.10 Minnesota Department of Health, 4740.2065, *Standard Operating Procedures*, Statutory Authority: MS s 144.97; 144.98; History: 31 SR 446, Posted: October 09, 2006, Revised April 16, 2010.

22) Appendix

22.1 Tables

Table 1: Instrument Tune Check Ion Abundance Criteria (TO-15)

Table 1A: Instrument Tune Check Ion Abundance Criteria (TO-14A)

Table 2: Volatile Organic Compounds, EPA Compendium Method TO-15 (SCAN)

Table 2A: Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)

Table 3: Standard Concentrations (SCAN) (Primary Sources)

Table 3A: Standard Concentrations (SIM) (Primary Sources)

Table 4: Standard Concentrations (SCAN) (Secondary Sources)

Table 4A: Standard Concentrations (SIM) (Secondary Sources)

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Daily QC and Sample Review Checklists

Attachment 4 – State and Project Specific Requirements

TABLE 1

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-15

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1] All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 1A

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-14A

Mass	Ion Abundance Criteria
50	15 to 40 percent of m/e 95
75	30 to 60 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5 to 9 Percent of m/e 95
173	Less than 2 Percent of m/e 174
174	>50 Percent of m/e 95
175	5 to 9 Percent of m/e 174
176	>95 and <101 Percent of m/e 174
177	5 to 9 Percent of m/e 176

Note: The criteria listed in Tables 1 and 1A shall be met or exceeded in order for EPA Compendium Methods TO-15 or TO-14A to be referenced.

TABLE 2 - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Bromochloromethane (IS1)	74-97-5	-	-	130	128, 132	-	-	-
Propene	115-07-1	42.08	NA	42	39,41	0.50	0.14	IS1
Dichlorodifluoromethane (CFC 12)	75-71-8	120.9	1.329	85	87, 101, 103	0.50	0.17	IS1
Chloromethane	74-87-3	50.49	0.911	50	52	0.50	0.15	IS1
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	76-14-2	170.9	1.455	135	137	0.50	0.19	IS1
Vinyl Chloride	75-01-4	62.50	0.9106	62	64	0.50	0.17	IS1
1,3-Butadiene	106-99-0	54.09	0.6149	54	39, 53	0.50	0.22	IS1
Bromomethane	74-83-9	94.94	1.6755	94	96	0.50	0.19	IS1
Chloroethane	75-00-3	64.52	0.8902	64	66	0.50	0.17	IS1
Ethanol	64-17-5	46.07	0.7893	45	46	5.0	0.80	IS1
Acetonitrile	75-05-8	41.05	0.7857	41	40	0.50	0.18	IS1
Acrolein	107-02-8	56.06	0.840	56	55	2.0	0.17	IS1
Acetone	67-64-1	58.08	0.7845	58	43	5.0	0.77	IS1
Trichlorofluoromethane	75-69-4	137.4	NA	101	103	0.50	0.17	IS1
Isopropyl Alcohol	67-63-0	60.10	0.7809	45	43	5.0	0.42	IS1
Acrylonitrile	107-13-1	53.06	0.8060	53	52	0.50	0.17	IS1
1,1-Dichloroethene	75-35-4	96.94	1.213	96	61	0.50	0.17	IS1
tert-Butanol	75-65-0	74.12	0.7887	59	57,41,43	1.0	0.33	IS1
Methylene Chloride	75-09-2	84.94	1.3266	84	49	0.50	0.17	IS1
Allyl Chloride	107-05-1	76.53	0.9376	41	76	0.50	0.16	IS1
Trichlorotrifluoroethane	76-13-1	187.38	1.5635	151	101	0.50	0.17	IS1

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Carbon Disulfide	75-15-0	76.14	1.2632	76	78	5.0	0.15	IS1
trans-1,2-Dichloroethene	156-60-5	96.94	1.2565	61	96	0.50	0.19	IS1
1,1-Dichloroethane	75-34-3	98.96	1.1757	63	65	0.50	0.16	IS1
Methyl tert-Butyl Ether	1634-04-4	88.15	0.7402	73	57	0.50	0.17	IS1
Vinyl Acetate	108-05-4	86.09	0.9317	86	43	5.0	0.65	IS1
2-Butanone (MEK)	78-93-3	72.11	0.7999	72	43	5.0	0.21	IS1
cis-1,2-Dichloroethene	156-59-2	96.94	1.2837	61	96	0.50	0.16	IS1
Diisopropyl Ether	108-20-3	102.18	0.7241	87	45,59,43	0.50	0.19	IS1
Ethyl Acetate	141-78-6	88.106	0.9003	61	70	1.0	0.35	IS1
n-Hexane	110-54-3	86.18	0.6548	57	86	0.50	0.15	IS1
Chloroform	67-66-3	119.4	1.4832	83	85	0.50	0.17	IS1
1,2-Dichloroethane-d4(S)	17060-07-0	-	-	65	67	-	-	IS1
Tetrahydrofuran	109-99-9	72.11	0.8892	72	71,42	0.50	0.20	IS1
Ethyl tert-Butyl Ether	637-92-3	102.176	0.7519	87	59,57	0.50	0.18	IS1
1,2-Dichloroethane	107-06-2	98.96	1.2351	62	64	0.50	0.16	IS1
1,4-Difluorobenzene(IS2)	540-36-3	-	-	114	88	-	-	-
1,1,1-Trichloroethane	71-55-6	133.4	1.3390	97	99, 61	0.50	0.17	IS2
Isopropyl acetate	108-21-4	102.13	0.8718	61	87,43	1.0	0.32	IS2
1-Butanol	71-36-3	74.1224	0.8098	56	41	1.0	0.48	IS2
Benzene	71-43-2	78.11	0.8765	78	77	0.50	0.16	IS2
Carbon Tetrachloride	56-23-5	153.8	1.5940	117	119	0.50	0.15	IS2

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Cyclohexane	110-82-7	84.16	0.7739	84	69,56	1.0	0.29	IS2
tert-Amyl Methyl Ether	994-05-8	102.176	0.7703	73	87,55,43	0.50	0.15	IS2
1,2-Dichloropropane	78-87-5	113	1.1560	63	62	0.50	0.16	IS2
Bromodichloromethane	75-27-4	163.8	1.980	83	85	0.50	0.15	IS2
Trichloroethene	79-01-6	131.4	1.4642	130	132	0.50	0.14	IS2
1,4-Dioxane	123-91-1	88.11	1.0337	88	58	0.50	0.16	IS2
Isooctane	540-84-1	114.23	0.6877	57	41	0.50	0.15	IS2
Methyl Methacrylate	80-62-6	100.12	0.944	100	69	1.0	0.31	IS2
n-Heptane	142-82-5	100.2	0.6837	71	57,100	0.50	0.17	IS2
cis-1,3-Dichloropropene	10061-01-5	111	1.224	75	77	0.50	0.14	IS2
4-Methyl-2-Pentanone	108-10-1	100.2	0.7965	58	85	0.50	0.16	IS2
trans-1,3-Dichloropropene	10061-02-6	111	1.217	75	77	0.50	0.16	IS2
1,1,2-Trichloroethane	79-00-5	133.4	1.4397	97	83	0.50	0.16	IS2
Chlorobenzene-d5(IS3)	3114-55-4	-	-	82	117	-	-	-
Toluene-d8(S)	2037-26-5	-	-	98	100	-	-	IS3
Toluene	108-88-3	92.14	0.8669	91	92	0.50	0.17	IS3
2-Hexanone	591-78-6	100.16	0.8113	43	58	0.50	0.16	IS3
Dibromochloromethane	124-48-1	208.3	2.451	129	127	0.50	0.16	IS3
1,2-Dibromoethane	106-93-4	187.9	2.1791	107	109	0.50	0.16	IS3
n-Butyl Acetate	123-86-4	116.16	0.8825	43	56, 73	0.50	0.16	IS3
n-Octane	111-65-9	114.23	0.6986	57	114	0.50	0.18	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Tetrachloroethene	127-18-4	165.8	1.6227	166	164	0.50	0.14	IS3
Chlorobenzene	108-90-7	112.6	1.1058	112	114	0.50	0.16	IS3
Ethylbenzene	100-41-4	106.2	0.8670	91	106	0.50	0.16	IS3
m-, p-Xylenes	179601-23-1	106.2	0.8642, 0.8611	91	106	1.0	0.30	IS3
Bromoform	75-25-2	252.8	2.899	173	175	0.50	0.15	IS3
Styrene	100-42-5	104.1	0.9060	104	78, 103	0.50	0.15	IS3
o-Xylene	95-47-6	106.2	0.8802	91	106	0.50	0.15	IS3
n-Nonane	111-84-2	128.26	0.7176	43	57, 85	0.50	0.15	IS3
1,1,2,2-Tetrachloroethane	79-34-5	167.9	1.5953	83	85	0.50	0.15	IS3
4-Bromofluorobenzene(S)	460-00-4	-	-	174	176	-	-	IS3
Cumene	98-82-8	120.2	0.8618	105	120	0.50	0.15	IS3
alpha-Pinene	80-56-8	136.24	0.8582	93	77	0.50	0.14	IS3
n-Propylbenzene	103-65-1	120.1938	0.8670	91	120,65	0.50	0.16	IS3
3-Ethyltoluene	620-14-4	120.2	0.8645	105	120	0.50	0.15	IS3
4-Ethyltoluene	622-96-8	120.2	0.8614	105	120	0.50	0.16	IS3
1,3,5-Trimethylbenzene	108-67-8	120.2	0.8652	105	120	0.50	0.16	IS3
alpha-Methylstyrene	98-83-9	118.19	0.9106	118	103,117	0.50	0.15	IS3
2-Ethyltoluene	611-14-3	120.2	0.8807	105	120	0.50	0.15	IS3
1,2,4-Trimethylbenzene	95-63-6	120.2	0.8758	105	120	0.50	0.15	IS3
n-Decane	124-18-5	142.28	0.7300	57	71,85	0.50	0.16	IS3
Benzyl Chloride	100-44-7	126.59	1.1004	91	126	0.50	0.11	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m³)	MDL[3] (µg/m³)	IS[4]
1,3-Dichlorobenzene	541-73-1	147	1.2884	146	148	0.50	0.15	IS3
1,4-Dichlorobenzene	106-46-7	147	1.2475	146	148	0.50	0.14	IS3
sec-Butylbenzene	135-98-8	134.2206	0.8601	105	134,91	0.50	0.16	IS3
p-Isopropyltoluene	99-87-6	134.2206	0.8573	119	134,91	0.50	0.15	IS3
1,2,3-Trimethylbenzene	526-73-8	120.1938	0.8944	105	120	0.50	0.15	IS3
1,2-Dichlorobenzene	95-50-1	147	1.3059	146	148	0.50	0.15	IS3
d-Limonene	5989-27-5	136.24	0.8402	68	93	0.50	0.14	IS3
1,2,Dibromo-3-Chloropropane	96-12-8	236.33	2.093	157	75, 39	0.50	0.099	IS3
n-Undecane	1120-21-4	156.31	0.7402	57	71, 85	0.50	0.15	IS3
1,2,4-Trichlorobenzene	120-82-1	181.5	1.459	180	182, 184	0.50	0.16	IS3
Naphthalene	91-20-3	128.17	1.0253	128	129	0.50	0.18	IS3
n-Dodecane	112-40-3	170.34	0.7487	57	71,85	0.50	0.13	IS3
Hexachlorobutadiene	87-68-3	260.8	1.556	225	227	0.50	0.14	IS3
Cyclohexanone	108-94-1	98.14	0.9478	55	42, 98	0.50	0.12	IS3
tert-Butylbenzene	98-06-6	134.22	0.867	119	134	0.50	0.15	IS3
n-Butylbenzene	104-51-8	134.22	0.867	91	134	0.50	0.17	IS3

(S) = Surrogate (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
NA = Not Available

Note 1: Additional compounds may be reported as long as the minimum requirements of this document are met. The compounds listed in this table are reported using TO-15 SCAN. The Selected Ion Monitoring (SIM) compounds are a subset of this list and are included in Table 2A.

Note 2: These are suggested primary and secondary ions. However, any ions in the analyte spectra that are sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 3: The laboratory performs three concentration level analyses (SIM, SCAN and Low Level SCAN). The method reporting limit listed is the standard SCAN limit (at or above lowest concentration in the initial calibration curve), but may change with each new initial calibration performed. Therefore, current reporting limits for the three analysis levels, MRLs in ppbv, and those from the Low Level SCAN should be reviewed in the electronic TO-15 Method Manual.

Note 4: The listing of the internal standard by which the compounds are quantitated is for TO-15 SCAN only. SIM compounds (SCAN subset) and their corresponding ions and internal standards are listed in Table 2A.

Note 5: m/e 101 is ~10% or less of m/e 85 (the base peak) and may not be present for low level results. Retention times must be carefully verified.

Table 2A – Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)					
Compound	**Primary Ion[1]**	**Secondary Ion[1]**	**MRL[2] (ug/m3)**	**MDL[2] (ug/m3)**	**IS**
Dichlorodifluoromethane	85	87	0.025	0.017	IS1
Chloromethane	52	50	0.025	0.019	IS1
Vinyl Chloride	62	64	0.025	0.0076	IS1
1,3-Butadiene	54	39	0.025	0.014	IS1
Bromomethane	94	96	0.025	0.0093	IS1
Chloroethane	64	66	0.025	0.0085	IS1
Acrolein	56	55	0.20	0.039	IS1
Acetone	58	43	2.5	0.056	IS1
Freon 11	101	103	0.025	0.015	IS1
1,1-Dichloroethene	96	98,61	0.025	0.0086	IS1
Methylene Chloride	84	49	0.10	0.013	IS1
Trichlorotrifluoroethane	151	153	0.025	0.0089	IS1
trans-1,2-Dichloroethene	96	98,61	0.025	0.0073	IS1
1,1-Dichloroethane	63	65	0.025	0.0061	IS1
Methyl tert-Butyl Ether	73	57	0.025	0.0093	IS1
cis-1,2-Dichloroethene	96	98,61	0.025	0.0092	IS1
Chloroform	83	85	0.10	0.018	IS1
1,2-Dichloroethane	62	64	0.025	0.0084	IS1
1,1,1-Trichloroethane	97	99	0.025	0.0059	IS1
Benzene	78	77	0.075	0.020	IS1
Carbon Tetrachloride	117	119	0.025	0.012	IS1
1,2-Dichloropropane	63	62,76	0.025	0.0073	IS2
Bromodichloromethane	83	85	0.025	0.0069	IS2
Trichloroethene	130	132	0.025	0.0085	IS2
1,4-Dioxane	88	58	0.10	0.0085	IS2
cis-1,3-Dichloropropene	75	77,39	0.025	0.0062	IS2
trans-1,3-Dichloropropene	75	77,39	0.025	0.0055	IS2
1,1,2-Trichloroethane	83	97,61	0.10	0.0079	IS2
Toluene	91	92	0.10	0.011	IS2
Dibromochloromethane	129	127	0.025	0.0088	IS3
1,2-Dibromoethane	107	109	0.025	0.0079	IS2
Tetrachloroethene	166	164	0.025	0.0082	IS2
Chlorobenzene	112	114	0.10	0.0092	IS3
Ethylbenzene	91	106	0.10	0.0097	IS3
m-&-p-Xylene	91	106	0.10	0.019	IS3
Styrene	104	103	0.10	0.0074	IS3
o-Xylene	91	106	0.10	0.0089	IS3
1,1,2,2-Tetrachloroethane	83	85	0.025	0.0072	IS3
1,3,5-Trimethylbenzene	105	120	0.10	0.0073	IS3
1,2,4-Trimethylbenzene	105	120	0.10	0.0083	IS3
1,3-Dichlorobenzene	146	148	0.025	0.0085	IS3
1,4-Dichlorobenzene	146	148	0.025	0.0081	IS3
1,2-Dichlorobenzene	146	148	0.025	0.0083	IS3
1,2-Dibromo-3-chloropropane	157	75	0.10	0.0095	IS3
1,2,4-Trichlorobenzene	182	184	0.025	0.013	IS3
Naphthalene	128	129	0.10	0.016	IS3
Hexachlorobutadiene	225	227	0.025	0.0092	IS3

NA = Not Available
(IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
Note 1: These are suggested primary and secondary ions. However, any ions in the analyte spectra that is sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 2: The method reporting limit listed is the standard SIM limit (lowest concentration in the initial calibration curve; must be higher than MDL), but may change with each new initial calibration performed. Therefore, current reporting limits should be reviewed. MDLs in ppbV may be reviewed in the electronic TO-15 Method Manual.

Table 3
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Bromochloromethane (IS1)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Propene	0.0792	0.198	0.396	0.99	4.95	24.75	49.5	99
Dichlorodifluoromethane (CFC 12)	0.0760	0.190	0.380	0.95	4.75	23.75	47.5	95
Chloromethane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
Vinyl Chloride	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
1,3-Butadiene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
Bromomethane	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
Chloroethane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
Ethanol	0.4128	1.032	2.064	5.16	25.80	129.00	258.0	516
Acetonitrile	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
Acrolein	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Acetone	0.4368	1.092	2.184	5.46	27.30	136.50	273.0	546
Trichlorofluoromethane	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Isopropyl Alcohol	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
Acrylonitrile	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,1-Dichloroethene	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
tert-Butanol	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
Methylene Chloride	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Allyl Chloride	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Trichlorotrifluoroethane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Carbon Disulfide	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
trans-1,2-Dichloroethene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,1-Dichloroethane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Methyl tert-Butyl Ether	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Vinyl Acetate	0.4200	1.050	2.100	5.25	26.25	131.25	262.5	525
2-Butanone (MEK)	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
cis-1,2-Dichloroethene	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
Diisopropyl Ether	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
Ethyl Acetate	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
n-Hexane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Chloroform	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
1,2-Dichloroethane-d4 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Tetrahydrofuran	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
Ethyl tert-Butyl Ether	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,2-Dichloroethane	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,4-Difluorobenzene(IS2)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
1,1,1-Trichloroethane	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
Isopropyl acetate	0.1832	0.458	0.916	2.29	11.45	57.25	114.5	229
1-Butanol	0.1824	0.456	0.912	2.28	11.40	57.00	114.0	228

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Benzene	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Carbon Tetrachloride	0.0920	0.230	0.460	1.15	5.75	28.75	57.5	115
Cyclohexane	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
tert-Amyl Methyl Ether	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
1,2-Dichloropropane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Bromodichloromethane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Trichloroethene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,4-Dioxane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Isooctane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Methyl Methacrylate	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
n-Heptane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
cis-1,3-Dichloropropene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
4-Methyl-2-Pentanone	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
trans-1,3-Dichloropropene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,1,2-Trichloroethane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Chlorobenzene-d5 (IS3)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene-d8 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
2-Hexanone	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
Dibromochloromethane	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
1,2-Dibromoethane	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
n-Butyl Acetate	0.0928	0.232	0.464	1.16	5.80	29.00	58.0	116
n-Octane	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
Tetrachloroethene	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
Chlorobenzene	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
Ethylbenzene	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
m- & p-Xylene	0.1728	0.432	0.864	2.16	10.80	54.00	108.0	216
Bromoform	0.0912	0.228	0.456	1.14	5.70	28.50	57.0	114
Styrene	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
o-Xylene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
n-Nonane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,1,2,2-Tetrachloroethane	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
4-Bromofluorobenzene (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Cumene	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
alpha-Pinene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
n-Propylbenzene	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
3-Ethyltoluene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
4-Ethyltoluene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,3,5-Trimethylbenzene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
alpha-Methylstyrene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
2-Ethyltoluene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,2,4-Trimethylbenzene	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
n-Decane	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
Benzyl Chloride	0.0912	0.228	0.456	1.14	5.70	28.50	57.0	114
1,3-Dichlorobenzene	0.0912	0.228	0.456	1.14	5.70	28.50	57.0	114
1,4-Dichlorobenzene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
sec-Butylbenzene	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
p-Isopropyltoluene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
1,2,3-Trimethylbenzene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,2-Dichlorobenzene	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
d-Limonene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
1,2-Dibromo-3-Chloropropane	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
n-Undecane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,2,4-Trichlorobenzene	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Naphthalene	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
n-Dodecane	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Hexachlorobutadiene	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
Methacrylonitrile	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Cyclohexanone	0.0944	0.236	0.472	1.18	5.90	29.50	59.0	118
tert-Butylbenzene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
n-Butylbenzene	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 3A - Standard Concentrations (SIM) (Primary Sources)[1]

Compound Name	10pg	20pg	50pg	100pg	500pg	1000pg	2500pg	10,000pg	20,000pg	50,000pg
Freon-12	9.50	19.00	47.50	95.0	475	950	2375	9500	19000	47500
Chloromethane	10.10	20.20	50.50	101.0	505	1010	2525	10100	20200	50500
Vinyl Chloride	10.00	20.00	50.00	100.0	500	1000	2500	10000	20000	50000
1,3-Butadiene	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
Bromomethane	10.20	20.40	51.00	102.0	510	1020	2550	10200	20400	51000
Chloroethane	10.10	20.20	50.50	101.0	505	1010	2525	10100	20200	50500
Acrolein	11.30	22.60	56.50	113.0	565	1130	2825	11300	22600	56500
Acetone	54.60	109.20	273.00	546.0	2730	5460	13650	54600	109200	273000
Freon-11	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
1,1-Dichloroethene	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
Methylene Chloride	11.30	22.60	56.50	113.0	565	1130	2825	11300	22600	56500
Freon-113	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
trans-1,2-Dichloroethene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,1-Dichloroethane	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
Methyl tert-Butyl Ether	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
cis-1,2-Dichloroethene	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
Chloroform	11.20	22.40	56.00	112.0	560	1120	2800	11200	22400	56000
1,2-Dichloroethane	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
1,1,1-Trichloroethane	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
Benzene	11.30	22.60	56.50	113.0	565	1130	2825	11300	22600	56500
Carbon Tetrachloride	11.50	23.00	57.50	115.0	575	1150	2875	11500	23000	57500
1,2-Dichloropropane	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
Bromodichloromethane	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
Trichloroethene	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
1,4-Dioxane	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
cis-1,3-Dichloropropene	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
trans-1,3-Dichloropropene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,1,2-Trichloroethane	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
Toluene	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
Dibromochloromethane	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
1,2-Dibromoethane	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
Tetrachloroethene	10.10	20.20	50.50	101.0	505	1010	2525	10100	20200	50500
Chlorobenzene	11.10	22.20	55.50	111.0	555	1110	2775	11100	22200	55500
Ethylbenzene	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
m,p-Xylenes	21.60	43.20	108.00	216.0	1080	2160	5400	21600	43200	108000
Styrene	11.20	22.40	56.00	112.0	560	1120	2800	11200	22400	56000
o-Xylene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,1,2,2-Tetrachloroethane	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
1,3,5-Trimethylbenzene	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
1,2,4-Trimethylbenzene	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
1,3-Dichlorobenzene	11.40	22.80	57.00	114.0	570	1140	2850	11400	22800	57000
1,4-Dichlorobenzene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,2-Dichlorobenzene	11.10	22.20	55.50	111.0	555	1110	2775	11100	22200	55500
1,2-Dibromo-3-chloropropane	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
1,2,4-Trichlorobenzene	11.30	22.60	56.50	113.0	565	1130	2825	11300	22600	56500
Naphthalene	11.10	22.20	55.50	111.0	555	1110	2775	11100	22200	55500
Hexachloro-1,3-butadiene	11.20	22.40	56.00	112.0	560	1120	2800	11200	22400	56000

Note 1: The concentrations detailed in Table 3A may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4 - Standard Concentrations (SCAN) (Secondary Sources)[1]

Compound Name	25ng	Compound Name	25ng	Compound Name	25ng
Bromochloromethane (IS1)	**25.0**	1,1,1-Trichloroethane	26.00	alpha-Pinene	26.00
Propene	25.00	Isopropyl acetate	54.50	n-Propylbenzene	25.25
Dichlorodifluoromethane (CFC 12)	25.50	1-Butanol	55.75	3-Ethyltoluene	26.50
Chloromethane	24.75	Benzene	27.50	4-Ethyltoluene	26.50
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	25.75	Carbon Tetrachloride	26.75	1,3,5-Trimethylbenzene	26.50
Vinyl Chloride	25.25	Cyclohexane	52.75	alpha-Methylstyrene	26.00
1,3-Butadiene	26.75	tert-Amyl Methyl Ether	26.25	2-Ethyltoluene	26.25
Bromomethane	25.25	1,2-Dichloropropane	26.50	1,2,4-Trimethylbenzene	26.25
Chloroethane	25.25	Bromodichloromethane	27.00	n-Decane	25.75
Ethanol	127.25	Trichloroethene	26.00	Benzyl Chloride	27.25
Acetonitrile	25.50	1,4-Dioxane	27.25	1,3-Dichlorobenzene	27.25
Acrolein	26.75	Isooctane	26.00	1,4-Dichlorobenzene	26.50
Acetone	135.00	Methyl Methacrylate	52.50	sec-Butylbenzene	26.75
Trichlorofluoromethane	24.75	n-Heptane	26.75	p-Isopropyltoluene	25.25
Isopropyl Alcohol	52.50	cis-1,3-Dichloropropene	28.25	1,2,3-Trimethylbenzene	26.25
Acrylonitrile	26.00	4-Methyl-2-Pentanone	27.25	1,2-Dichlorobenzene	26.75
1,1-Dichloroethene	26.75	trans-1,3-Dichloropropene	27.00	d-Limonene	26.25
tert-Butanol	52.75	1,1,2-Trichloroethane	26.50	1,2-Dibromo-3-Chloropropane	25.75
Methylene Chloride	27.00	**Chlorobenzene-d5 (IS3)**	**25.0**	n-Undecane	25.25
Allyl Chloride	27.25	**Toluene-d8 (S)**	**25.0**	1,2,4-Trichlorobenzene	26.25
Trichlorotrifluoroethane	27.00	Toluene	26.50	Naphthalene	24.50
Carbon Disulfide	24.50	2-Hexanone	27.75	n-Dodecane	25.25
trans-1,2-Dichloroethene	26.50	Dibromochloromethane	27.50	Hexachlorobutadiene	26.75
1,1-Dichloroethane	26.00	1,2-Dibromoethane	27.00	Methacrylonitrile	26.00
Methyl tert-Butyl Ether	26.50	Butyl Acetate	28.00	Cyclohexanone	27.75
Vinyl Acetate	128.00	n-Octane	26.00	tert-Butylbenzene	26.50
2-Butanone (MEK)	27.00	Tetrachloroethene	24.75	n-Butylbenzene	27.25
cis-1,2-Dichloroethene	26.75	Chlorobenzene	27.00		
Diisopropyl Ether	27.25	Ethylbenzene	26.50		
Ethyl Acetate	53.50	m- & p-Xylene	52.50		
n-Hexane	26.25	Bromoform	27.00		
Chloroform	27.00	Styrene	27.25		
1,2-Dichloroethane-d4 (S)	**25.0**	o-Xylene	25.75		
Tetrahydrofuran	25.75	n-Nonane	25.50		
Ethyl tert-Butyl Ether	26.50	1,1,2,2-Tetrachloroethane	25.25		
1,2-Dichloroethane	26.25	**4-Bromofluorobenzene (S)**	**25.0**		
1,4-Difluorobenzene(IS2)	**25.0**	Cumene	25.50		

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4A – ICV/LCS Standard Concentrations (SIM) (Secondary Sources)[1]

Compound Name	500pg
Freon-12	510
Chloromethane	495
Vinyl Chloride	505
1,3-Butadiene	535
Bromomethane	505
Chloroethane	505
Acrolein	535
Acetone	2700
Freon-11	495
1,1-Dichloroethene	535
Methylene Chloride	540
Freon-113	540
trans-1,2-Dichloroethene	530
1,1-Dichloroethane	520
Methyl tert-Butyl Ether	530
cis-1,2-Dichloroethene	535
Chloroform	540
1,2-Dichloroethane	525
1,1,1-Trichloroethane	520
Benzene	550
Carbon Tetrachloride	535
1,2-Dichloropropane	530
Bromodichloromethane	540
Trichloroethene	520
1,4-Dioxane*	545
cis-1,3-Dichloropropene	565
trans-1,3-Dichloropropene	540
1,1,2-Trichloroethane	530
Toluene	530
Dibromochloromethane	550
1,2-Dibromoethane	540
Tetrachloroethene	495
Chlorobenzene	540
Ethylbenzene	530
m,p-Xylenes	1050
Styrene	545
o-Xylene	515
1,1,2,2-Tetrachloroethane	505
1,3,5-Trimethylbenzene	530
1,2,4-Trimethylbenzene	525
1,3-Dichlorobenzene	545
1,4-Dichlorobenzene	530
1,2-Dichlorobenzene	535
1,2-Dibromo-3-chloropropane	515
1,2,4-Trichlorobenzene	525
Naphthalene	490
Hexachloro-1,3-butadiene	535

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Attachment 1

Training Plan

Training Plan for Analysis of VOCs by GC/MS

Trainee _____ Trainer _____ Instrument _____ Training Completion Date _____

1. Read SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____

2. Read Methods TO-14A & TO-15A *Training Duration* _____ Trainer ____ Trainee ____ Date _____

3. Demonstrated understanding of the scientific basis of the analysis Trainer ____ Trainee ____ Date _____
   Whole air sample preconcentration techniques
   Gas chromatography *Training Duration* _____
   Mass spectrometry

4. Demonstrated familiarity with related SOPs Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences; Rev. ___
   SOP for Making Entries onto Analytical Records; Rev. ___ *Training Duration* _____
   SOP for Manual Integration Policy; Rev. ____
   SOP for Significant Figures; Rev. ____
   SOP for Nonconformance and Corrective Action; Rev. ____
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation; Rev. ____
   SOP for Cleaning and Certification of Summa Canisters; Rev. ____

5. Observe performance of SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant introduction (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

6. Perform SOP with supervision *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant use (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

7. Independent performance of the SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant proficiency (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)
   ___initial demonstration of competency (4 Laboratory Control Samples)

8. Instrument operation and maintenance Trainer ____ Trainee ____ Date _____
   ___autosampler *Training Duration* _____
   ___GC and capillary column installation *Training Duration* _____
   ___mass spectrometer *Training Duration* _____
   ___data system *Training Duration* _____

Attachment 2

Initial Calibration Checklist

Initial Calibration Review Checklist - EPA Compendium Method TO-15

ICAL Date:_____ ICAL ID:_____ LIMS ICAL ID:_____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS11 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan Scan Low Level (0.1ng): ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation in the ICAL file?	☐
	☐ BFB Tune analysis Report	☐
	☐ Calibration Status Report (aka Calibration History)	☐
	☐ Response Factor Report/Percent RSD	☐
	☐ Quantitation Report for each calibration standard (including manual integration documentation)	☐
	☐ ICV Quantitation Report	☐
	☐ TO-15 Standard Calculation Spreadsheet	☐
☐	2. Was the ICAL performed continuously (not interrupted for maintenance or sample analysis)?	☐
☐	3. Have all the calibration standards been analyzed within 24 hours of each other?	☐
☐	4. Does the BFB tune check standard analysis at the start meet the tune criteria?	☐
☐	5. Are all the analytes in the blank analysis <MRL?	☐
☐	6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels?	☐
☐	7. Were the standards analyzed from low concentration to high concentration?	☐
☐	8. For each analyte, are there no levels skipped?	☐
☐	9. For each analyte, is there only one value used for each calibration level?	☐
☐	10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL?	☐
☐	11. For each analyte, is the corresponding signal to noise ratio at least 3:1 at the lowest point on the curve?	☐
☐	12. For each analyte, are the corresponding upper levels free from saturation?	☐
☐	13. If a calibration level is dropped, are all the responses for each target analyte dropped and is the information noted in the ICAL explaining the reason?	☐
☐	14. Is the average RSD ≤30% for all analytes, with no more than two exceptions ≤40%?	☐
☐	15. Is the response Y at each calibration level within 40% of the mean area response over the initial calibration range for each internal standard?	☐
☐	16. Percent recovery for each analyte in the ICV 70%-130% (50-150% for VA, unless AFCEE or DoD)?	☐
☐	17. Was the RRT for each target compound at each calibration level within 0.06RRT units of the mean RRT for the compound?	☐
☐	18. Is the retention time shift for each of the internal standards at each calibration level within 20s of the mean retention time over the initial calibration range for each standard?	☐
☐	19. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages.	☐
☐	20. Is the ICAL good at 0.5ng (or 0.1ng)–100ng (Scan) or 10-20000pg (SIM) for all compounds? ☐ Yes ☐ No If not, note exceptions and the corresponding MRLs below – *Specify applicable range...*	☐
☐	21. Are ALL of the peak selections for each analyte correct according to retention time (all RTs must be checked by both the initial and peer reviewer)?	☐

COMMENTS:

Analyst:_____ Date: _____

Secondary Reviewer: _____ Date: _____

Attachment 3

Daily QC and Sample Review Checklists

Daily QC Review Checklist

(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

EPA Compendium Method TO-15

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation present? ☐ CORRECT BFB Tune analysis Report ☐ CCV analysis Quantitation Report & %D Report ☐ LCS analysis Quantitation Report ☐ MB analysis Quantitation Report	☐
☐	2. BFB **tune** check standard analysis meet the tune criteria for the method indicated above?	☐
☐	3. Analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**?	☐
☐	4. Does the **CCV** have a difference ≤30% for all analytes? [Note <u>all</u> outliers biased high and/or low]	☐
☐	5. All **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)?	☐
☐	6. All **IS** responses within ±40% of CCV or the midpoint in the ICAL?	☐
☐	7. All **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%)	☐
☐	8. All analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)?	☐
☐	9. **LCS** %R within the lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)?	☐
☐	10. All analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits?	☐

COMMENTS:

Air-Phase Petroleum Hydrocarbons

☐	1. Does the **CCV** meet the following criteria? • Percent difference ≤30%. • One compound or range can be >30%, but less than 50%. • No single analyte or range may be >50%. [Note outliers biased high and/or low]	☐
☐	2. Does lab **duplicate** meet an RPD of ≤30% for results >5x MRL? Repeat analysis if:	☐
☐	3. Are the analytes in the **LCS** within 70%-130% recovery?	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐ LIMS Run Approval ☐ LIMS Supervisor Approval

Analyst: _____ Secondary Reviewer: _____

Date: _____ Date: _____

Sample Review Checklist
(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

EPA Compendium Method TO-15

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____ **Project #:** _____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. All analyte hits in the samples within the **calibration range** and/or noted?	☐
☐	2. All **peak integrations** acceptable?	☐
☐	3. All **manual integrations** flagged and documented?	☐
☐	4. Have **Q values** been verified for each peak?	☐
☐	6. All **calculations** correct?	☐
☐	7. Has the analyst initialed and dated each **quantitation report**?	☐
☐	8. For **TICs** are the relative intensity and other requirements met?	☐
☐	9. **Auto report** correct?	☐
☐	10. **MRL** = _____ ☐ ng ☐ pg (ethanol, acetone, vinyl acetate = 5.0ng)	☐
☐	11. Pressurized with **Helium**? Is the worksheet completed for all samples?	☐
☐	12. Report to **MDL**? ☐ Yes ☐ No	☐
☐	13. **Global Minimum Detection Limit** = _____ ☐ ng ☐ pg	☐
☐	14. **DOD:** Are **manual integrations** notated in the **case narrative?**	☐

COMMENTS:

Air-Phase Petroleum Hydrocarbons

☐	1. Are all manual **integrations** flagged and documented (except for HC ranges)?	☐
☐	2. Are all peak **integrations** acceptable?	☐
☐	3. Has the analyst initialed and dated each **quantitation report**?	☐
☐	4. Are the associated ICAL responses correct?	☐
☐	5. Are the sample responses entered into the template correctly?	☐
☐	6. Are the TO-15 target compounds entered into the template correctly?	☐
☐	7. Does the lab **duplicate** meet a RPD of ≤30% for results >5x the MRL? Otherwise, repeat analyses if:	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐ LIMS Run Approval

Analyst: _____

Date: _____

☐ LIMS Supervisor Approval

Secondary Reviewer: _____

Date: _____

Attachment 4

State and Project Specific Requirements

Minnesota Requirements	
Item	**Criteria**
Holding Time (HT)	14 days
Tedlar bags	Not allowed for sampling or sample dilution
Canisters and flow controllers	Individually certified Individually leak checked before shipment
	Samples with concentrations outside of the calibration curve will have a zero canister analysis performed to check for carryover. If carryover is detected, system bake out shall be performed and documented. Additionally, in instances where the laboratory has evidence on file that a particular compound when present at a high concentration does not exhibit carry-over, the samples will not be reanalyzed. When samples are analyzed that have a higher concentration than the evidence on file, the above requirements must be followed. Also, samples that have hits below the MRL will not be reanalyzed when analyzed after a sample with concentrations over the calibration range.
Method Reporting Verification Check	Analyze a Method Reporting Verification at the beginning of the sequence prior to analyzing samples. Acceptance criteria ±40%.
Duplicates	10 percent laboratory duplicates
Record retention	MN/NELAC 5 years MPCA (Minnesota Pollution Control Agency) compliant samples 10 years
Tier level	TIII

Arizona Requirements	
Item	**Criteria**
LCS	70-130% (vinyl acetate 50-150%)

Department of Toxic Substances Control (DTSC) Requirements	
Item	**Criteria**
Holding Time (HT)	72 hour hold time for canisters

EPA Region 9 Requirements	
Item	**Criteria**
Holding Time (HT)	14 days

ATTACHMENT D

QUALITY CONTROL FORMS

FIELD ASSESSMENT CHECKLIST

		Yes	No	NA	Explain
A	**GENERAL PROCEDURES**				
A1	**PROJECT PLANS**				
	Is a copy of the approved Work Plan (WP) onsite and readily available to field personnel?				
	Is a copy of the approved Accident Prevention Plan (APP) onsite and readily available to field personnel?				
	Is a copy of the approved Quality Assurance Project Plan (QAPjP) onsite and readily available to field personnel?				
	Are field personnel knowledgeable of the project plans?				
A2	**ORGANIZATION, PERSONNEL, AND RESPONSIBILITIES**				
	Does either the WP or the QAPjP include an organizational chart?				
	Does either the WP or the QAPjP include a list of personnel responsibilities?				
	Is a daily meeting held to present the planned activities to the team and provide updates on any health and safety and Quality Control (QC)				
	Do the field personnel understand the chain of command? Discuss with individuals.				
	Have all field personnel reviewed and signed the APP?				
	Do field personnel have certification documentation on hand?				

		Yes	No	NA	Explain
	Is the site secure from unauthorized personnel?				
A3	**SAMPLE DOCUMENTATION AND HANDLING**				
	Is a Sample Manager/Custodian identified? Name:				
	Have all personnel involved with sample handling/shipment received training in all applicable and appropriate procedures?				
	Are all transfers of sample custody documented?				
	Have samples been preserved as specified in the approved QAPjP?				
	Does the identification and packaging of samples include each of the following items:				
	a. Entries in permanent ink of all applicable sample labels and tags, chain-of-custody (COC) forms and seals, and any associated paperwork?				
	b. Legible sample IDs				
	c. Complete and legible sample labels				
	d. Signed and dated COC forms				
	e. Placement of groundwater samples in a clear Ziploc plastic bag (if applicable)				
	Are sample collection logs and shipping records maintained onsite, well organized and accurate? Is any QC performed?				
	Are the coolers packed in a contamination-free area?				

		Yes	No	NA	Explain
A3	**SAMPLE DOCUMENTATION AND HANDLING (continued)**				
	Is there adequate protection against breakage of sample containers (for example packed with sufficient padding material)?				
	Are procedures in place to ensure that samples that need to be maintained at 4 degrees C are kept at that temperature and are not going to freeze or overheat?				
	Are all entries on the COC and associated paperwork (field forms, logbook, air bill) complete, accurate, legible and made in permanent ink where required?				
	Are samples shipped to the appropriate laboratories?				
	Are COC forms placed in a clear waterproof Ziploc plastic bag and taped to the inside of the cooler or box lid?				
	If coolers are shipped, are they properly secured with duct and clear/strapping tape?				
	Are shipping labels filled out properly?				
	Are shipping labels appropriately secured to the outside of the coolers? Additional clear tape is needed to secure self-adhesive air bill envelopes.				
	Are samples shipped to the laboratories in a timely fashion to minimize potential problems with holding times exceedances?				
	Is there a system in place to report sample shipments to the laboratory contact?				
	Is there a system in place to report sample shipments to project personnel not present onsite?				
	Are accurate sample collection logs and shipping records maintained onsite in a well-organized fashion?				

		Yes	No	NA	Explain
A4 FIELD RECORDS					
	Are daily field activity logs used and kept onsite?				
	Are all field activity logs dated and signed?				
	Are all entries in the log made promptly? Is time indicated in military format?				
	Are any blank pages or spaces left in the log? Any blank space in the log as well as the bottom of the last page should be crossed out, signed and dated.				
	Are all field log entries made in indelible ink?				
	Are all corrections indicated by a single-line strikethrough, dated and initialed?				
	Do all logs contain at least the following information on the cover:				
	a. Project name				
	b. Site name				
	c. Weather notes/visitors				
	d. Start and end dates for the field effort				
	e. Names of the individuals that are using the logbook?				
	Do all logs contain at least the following information on the cover:				
	Are entries in the logs adequate to allow a competent person other than the originator to reconstruct the activities?				
	Are sufficient data recorded to allow all field calculation to be replicated (for example total purge calculations for soil-vapor sampling)?				
	Are field calculations accurate? The inspector should verify 10% of the calculations.				
	Are soil-boring logs filled out promptly, accurately, and legibly?				

		Yes	No	NA	Explain
	Does sample collection information include the following items:				
	a. Sampling personnel				
	b. Sample identification				
	c. Sampling location map				
	d. Sample depth				
	e. Sample description				
	f. Collection date				
	g. Collection time (military time)				
	h. Ambient weather conditions				
	i. Analytical suite				
	j. Field data sheets				
	Are sample log sheets or electronic tracking file filled out promptly accurately and legibly?				
A5	**INSTRUMENT MAINTENANCE AND CALIBRATION**				
	Has all analytical and monitoring equipment been calibrated according to the schedule required in the approved WP or QAPjP?				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				

		Yes	No	NA	Explain
	Are all instruments In use (or that will be used today) within calibration tolerance? Inspector should request that the designated calibration personnel analyze the appropriate standard as an unknown.				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Are all instruments used properly, as detailed in the FSP or QAPjP and according to SOPs and manufacturer's instructions?				
	Are all instruments appropriately maintained, according to manufacturer's instructions?				
	Additional comments on section A.				
B	**FIELD PROCEDURES**				
B1	Has the inspector reviewed the WP and QAPjP prior to the site visit?				
B2	Do the WP or QAPjP include specific procedural instructions, necessary for the completion of the scheduled field activities?				
B3	Do the WP or QAPjP and SOPs provide sufficient detail and effectively describe the objectives and requirements of the activities?				
B4	Is all equipment necessary to complete the work at hand and in good working condition?				

		Yes	No	NA	Explain
B5	Are daily reports prepared to identify deviations from the approved plans or SOPs?				
B6	Have appropriate authorizations been granted on all the deviations?				
B7	Have all field and QC samples been collected as per the WP or QAPjP and applicable instructions? Inspector should check for proper sample collection order, for example volatile organic compounds (VOC) first, proper containers, no homogenization for samples to be analyzed for VOCs, etc.).				
B8	Were any samples collected in a fashion that may question their integrity; for example, VOCs collected in the vicinity of a running vehicle?				
B9	Additional comments on section B.				
C	**INSPECTION SUMMARY**				
C1	Do the responses to the inspector indicate that the field personnel are aware of the quality assure (QA)/QC and its importance to the success of the projects?				
C2	Do field personnel place positive emphasis on QA/QC procedures?				
C3	Have responses with respect to QA/QC procedures been open and direct?				
C4	Has a cooperative attitude been displayed by the field personnel?				
C5	Are all procedures and documentation performed consistent with the Work Plan or QAPjP? Is there evidence that the field team has corrected deficiencies identified in the previous inspection (if applicable)?				
C6	Additional comments on section C.				
D	**DEFICIENCY REPORT**				
D1	Were any deficiencies identified as a result of the field inspection?				
D2	Were all deficiencies and observations discussed with the field team leader during inspection debriefing?				

		Yes	No	NA	Explain
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				

		Yes	No	NA	Explain
D	**DEFICIENCY REPORT (continued)**				
D2	Were all deficiencies and observations discussed with the field team leader during inspection debriefing? (continued)				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				

		Yes	No	NA	Explain
D	**DEFICIENCY REPORT (Continued)**				
D2	Were all deficiencies and observations discussed with the field team leader during inspection debriefing? (Continued)				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				

		Yes	No	NA	Explain
D	**DEFICIENCY REPORT (Continued)**				
D2	Were all deficiencies and observations discussed with the field team leader during inspection debriefing? (Continued)				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				

NON-CONFORMANCE REPORT

Project:		RFI No.:		Date:
To:		Contract No:		
		REFERENCES		
Attention:		Drawing/Spec:		
Subject:		Detail/Section:		
		Discipline:		

POTENTIAL IMPACT	ROUTING	DATE SENT	DATE REC'D	COMMENTS
☐ QUALITY/TECHNICAL COMPLETION				
☐ COST				
☐ SCHEDULE				
ACTIVITY:				
	RESPONSE REQUESTED BY:		PRIORITY:	

NONCONFORMANCE	
CORRECTIVE ACTION	

Addressee: Sign and return original to:	**By:**
	Name/ Signature:
	Title:

WORK PLAN FOR

SOIL VAPOR MONITORING AND DRINKING WATER MONITORING

SOLID WASTE MANAGEMENT UNIT ST-106/SS-111

~~April~~ August 2016

377 MSG/CEANR
2050 Wyoming Blvd. SE
KIRTLAND AFB, NEW MEXICO 87117-5270

KIRTLAND AIR FORCE BASE
ALBUQUERQUE, NEW MEXICO

Work Plan for
Soil Vapor Monitoring and
Drinking Water Monitoring
Solid Waste Management Unit ST-106/
SS-111

~~April~~ August 2016

Prepared for

U.S. Army Corps of Engineers
Albuquerque District
Albuquerque, New Mexico 87109

USACE Contract No. W912PP-16-C-0002

Prepared by

Sundance Consulting, Inc.
8210 Louisiana Blvd, Suite C
Albuquerque, NM 87113

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	~~April~~ August 2016	Work Plan

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R. Hobbs, P.G.

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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Albuquerque, NM 87113

8. PERFORMING ORGANIZATION REPORT NUMBER

KAFB-16-002

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

USACE Albuquerque District

4101 Jefferson Plaza NE Albuquerque, NM 87109

Project Manager: Trent Simpler, P.E.

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

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12b. DISTRIBUTION CODE

13. This Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP) has been prepared by Sundance Consulting, Inc. as part of the ongoing monitoring effort at Solid Waste Management Unit (SWMU) ST-106/SS-111 at the Kirtland Air Force Base (KAFB) Bulk Fuels Facility. The WP demonstrates the U.S. Air Force's commitment to continuing with the treatment of the fuel contamination resulting from past practices and events at SWMU ST-106/SS-111. This WP outlines activities to be performed in support of continued monitoring of drinking water and the nature and extent of soil vapor contamination at SWMU ST-106/SS-111, and in conjunction with the Quality Assurance Project Plan, will become the procedural guidance document for these activities. This document meets the most recent requirements of the Department of Defense (DoD) regarding planning documents for DoD facilities. The WP was written in accordance with KAFB's Resource Conservation and Recovery Act Permit Number NM9570024423.

14. SUBJECT TERMS

Bulk Fuels Facility (BFF), Soil Vapor Monitoring and Drinking Water Monitoring Work Plan

15. NUMBER OF PAGES

126

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DOCUMENT CERTIFICATION

~~APRIL~~ AUGUST 2016

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision according to a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowing violations.

_____

ERIC H. FROEHLICH, Colonel, US Air Force

Commander, 377th Air Base Wing

This document has been approved for public release.

_____

KIRTLAND AIR FORCE BASE

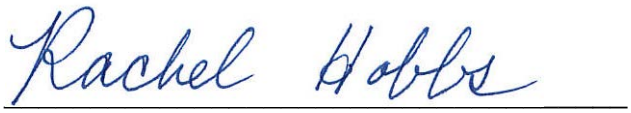
377th Air Base Wing Public Affairs

PREFACE

This Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP) was prepared by Sundance, Consulting, Inc. (Sundance) for the U.S. Army Corps of Engineers (USACE) under contract W912PP-16-C-0002. It pertains to the Kirtland Air Force Base (KAFB) Bulk Fuels Facility site at Solid Waste Management Unit ST-106/SS-111, located in Albuquerque, New Mexico. This WP was prepared in accordance with the permit issued to KAFB under the Resource Conservation and Recovery Act and applicable federal, state, and local laws and regulations.

This WP presents and describes all activities associated with the quarterly sampling of 284 soil vapor monitoring points and the monthly sampling of four drinking water production wells.

This WP is prepared for work to be performed between 20 January 2016 and 20 January 2018. Ms. Amy Sanchez is the Contracting Officer's Representative for the USACE Albuquerque District, and Mr. Trent Simpler, Professional Engineer, is the Project Manager. Mr. Wayne Bitner, Jr. is the KAFB Restoration Section Chief, ~~and Patrick Scher, Professional Geologist (P.G.), is the Sundance Project Manager.~~ This plan was prepared by ~~Senior Project Geologist~~ Rachel Hobbs, P.G, the Sundance Project Manager.



~~Patrick L. Scher,~~ Rachel Hobbs, P.G.

Sundance Consulting, Inc.

~~Senior~~ Project Manager

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ACRONYMS AND ABBREVIATIONS

%	percent
^{o}C	degrees Celsius
AFB	Air Force Base
AFCEC	Air Force Civil Engineering Center
Air Force	U.S. Air Force
ALS	ALS Environmental Laboratories
APH	air-phase petroleum hydrocarbons
APP	Accident Prevention Plan
BFF	Bulk Fuels Facility
BTEX	benzene, toluene, ethylbenzene, and xylenes
CFR	Code of Federal Regulations
CO_2	carbon dioxide
COA	City of Albuquerque
CY	calendar year
DO	dissolved oxygen
DoD	U.S. Department of Defense
DTIC	Defense Technical Information Center
EDB	ethylene dibromide
e.g.	example given
EPA	U.S. Environmental Protection Agency
ERP	Environmental Restoration Program
ERPIMS	Environmental Resource Program Information Management System
HC	hydrocarbons
Horiba	Horiba MEXA 584L auto emissions analyzer
IDW	investigation-derived waste
in Hg	inches of mercury
in WC	inches of water column
KAFB	Kirtland Air Force Base
LDC	Laboratory Data Consultants, Inc.
NMED	New Mexico Environment Department
O_2	oxygen
ORP	oxidation reduction potential
P.E.	Professional Engineer
P.G.	Professional Geologist
PID	photoionization detector
PPE	personal protective equipment
ppmv	parts per million by volume

QAPjP	Quality Assurance Project Plan
QC	quality control
QMR	Quarterly Monitoring and Site Investigation Report
RCRA	Resource Conservation and Recovery Act
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
Sundance	Sundance Consulting, Inc.
SVE	soil vapor extraction
SVEW	soil vapor extraction well
SVM	soil vapor monitoring
SVMP	soil vapor monitoring point
SWMU	Solid Waste Management Unit
SWMW	soil vapor monitoring well
U.S.	United States
USACE	U.S. Army Corps of Engineers
VA	Veteran's Administration
VOC	volatile organic compound
WP	Work Plan
YSI	Yellow Springs Instruments

EXECUTIVE SUMMARY

This Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP) has been prepared by Sundance Consulting, Inc. as part of the ongoing monitoring effort at Solid Waste Management Unit (SWMU) ST-106/SS-111 at the Kirtland Air Force Base (KAFB) Bulk Fuels Facility. The WP demonstrates the U.S. Air Force's commitment to continuing with the treatment of the fuel contamination resulting from past practices and events at SWMU ST-106/SS-111. This WP outlines activities to be performed in support of continued monitoring of drinking water and the nature and extent of soil vapor contamination at SWMU ST-106/SS-111, and in conjunction with the Quality Assurance Project Plan, will become the procedural guidance document for these activities. These documents meet the most recent requirements of the Department of Defense (DoD) regarding planning documents for DoD facilities. The WP was written in accordance with KAFB's Resource Conservation and Recovery Act Permit Number NM9570024423.

The objective of the WP is to detail the quarterly sampling and analysis of the existing soil vapor monitoring network, and monthly drinking water well sampling and analysis activities to be implemented. The work to be completed is presented under each of the tasks listed below:

- Perform quarterly sampling and reporting at 284 soil vapor monitoring points for 8 quarters beginning in first quarter calendar year (CY) 2016.
- Perform yearly maintenance of the soil vapor monitoring network.
- Abandon and install soil vapor monitoring locations.
- Perform monthly sampling and reporting of four drinking water production wells beginning in February 2016 through the end of CY 2017.

1 INTRODUCTION

This Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP) was prepared by Sundance, Consulting, Inc. (Sundance) for the U.S. Army Corps of Engineers (USACE) under contract W912PP-16-C-0002. The WP pertains to sampling activities at the Kirtland Air Force Base (KAFB) Bulk Fuels Facility (BFF), Solid Waste Management Unit (SWMU) ST-106/SS-111 (example given [e.g.] BFF site). Environmental restoration efforts at the BFF site are being conducted under requirements set forth in the Resource Conservation and Recovery Act (RCRA) Permit No. NM9570024423 with the New Mexico Environment Department (NMED) serving as the lead regulatory agency (NMED, 2010).

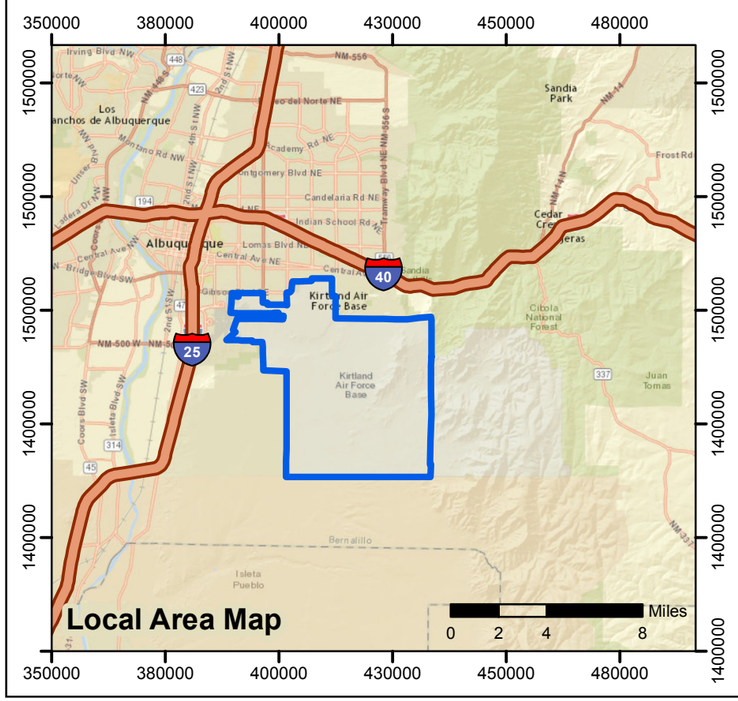
This WP addresses activities that are continuing the implementation of the RCRA process for this site, including the continued monitoring of the soil vapor plume, and monitoring of drinking water production wells. This WP will become the procedural guidance document for these activities to be performed as part of the ongoing investigation and will meet the most recent requirements of the Department of Defense (DoD) regarding planning documents for DoD facilities.

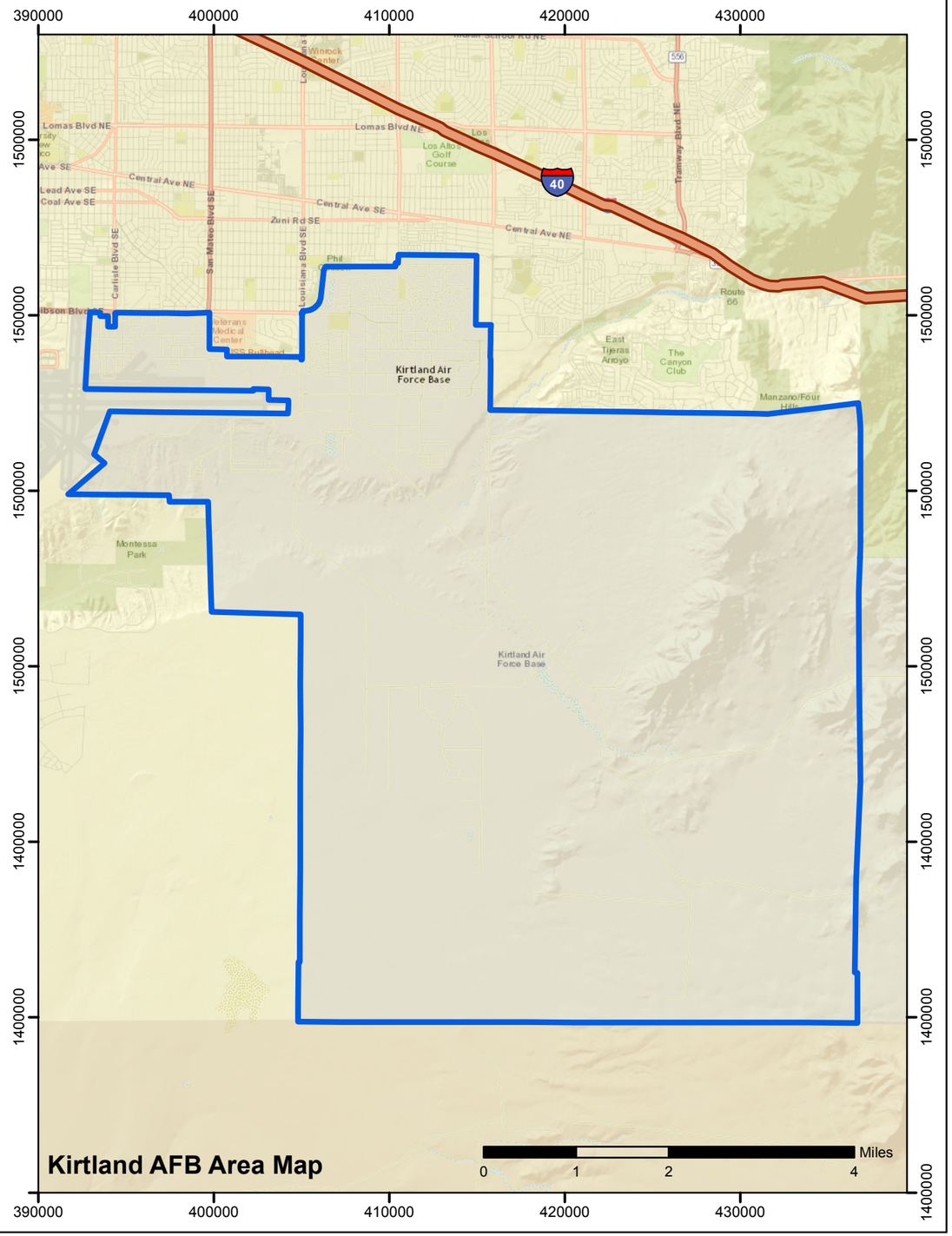
Requirements for the protection of health and safety on the job sites are addressed in the companion *Quarterly Soil Vapor Sampling and Monthly Drinking Water Sampling Accident Prevention Plan* (APP) (Sundance 2016a). The APP also incorporates the Site Safety and Health Plan (SSHP).

1.1 Overview and Scope of Activities

The BFF site is located in Albuquerque, New Mexico (Figure 1-1). Field activities presented in this WP include quarterly soil vapor sampling and analysis of 284 monitoring points installed as part of the investigation at SWMU ST-106/SS-111. In addition, monthly sampling and analysis of four drinking water production wells will be performed. Analytical results from soil vapor and drinking water samples will be reported in the Quarterly Monitoring and Site Investigation Reports (QMRs). Analytical results from the drinking water production well samples will be reported in the QMRs. This WP for quarterly soil vapor sampling and monthly drinking water sampling includes all of the elements of a Sampling and Analysis Plan/Field Sampling Plan and covers all the project tasks associated with:

- Sample soil vapor monitoring network quarterly, beginning in first quarter calendar year (CY) 2016 for eight (8) quarters.
- Sample drinking water production wells monthly, beginning in February 2016 through the end of CY 2017.
- Perform annual maintenance of the soil vapor monitoring network.
- Abandon and install soil vapor monitoring locations.
- Analyze soil vapor and drinking water production well samples, and report results quarterly.





Legend

- Interstate
- US Highway
- State/County Highway
- States
- Kirtland Air Force Base Installation Area

N

Credits: Esri, HERE, DeLorme, USGS, Intermap

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

Figure 1-1

Site Location Map
Work Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016

1.2 Report Organization

The BFF Soil Vapor Monitoring and Drinking Water Monitoring WP is divided into the following sections:

- Section 1—Presents an introduction to the plan, an overview of the project, and the scope of activities and organization of the WP.
- Section 2—Presents the BFF site description and operational history.
- Section 3—Project tasks are summarized with sufficient detail on how they will be accomplished.
- Section 4—Presents the project schedule.
- Section 5—Provides information on the organizational plan for the execution of work.
- Section 6—Refers to the data management requirements.
- Section 7—Presents information on the management and disposal of the waste generated during this project.
- Section 8—Presents references cited for this WP.

Associated appendices are provided at the end of this WP as follows:

- Appendix A: Field Forms

  -Field Activity Log

  -Well Integrity Checklist

  -Horiba Calibration Form

  -Leak Test Log

  -Example Soil Vapor Purge Log

  -Example Soil Vapor Sample Collection Log

  -Example Soil Vapor Chain of Custody

  -Example Water Sample Collection Log

  -Example Water Chain of Custody
- Appendix B: Project Schedule
- Appendix C: Quality Assurance Project Plan (QAPjP)

2 BACKGROUND INFORMATION

2.1 Site Description

KAFB is located in Bernalillo County, in central New Mexico, southeast of and adjacent to the City of Albuquerque (COA) and the Albuquerque International Sunport (Figure 1-1). The approximate area of the base is 52,287 acres. The BFF site is located in the northwestern portion of KAFB.

2.2 Site History

The BFF and associated infrastructure operated from 1953 through 1999. During this time, the fueling area was separated into a tank holding area where bulk shipments of fuel were received and a fuel off-loading area where individual fuel railcars or trucks were emptied. KAFB stopped using the underground piping at the facility in 1999 due to discovery of leaks in buried fuel transfer piping.

Even though a fuel leak was identified by KAFB in 1999, the exact history of the leaks or releases is unknown. Releases most likely occurred when fuel was transferred from railcars and trucks to the pump house. Initially, it was thought that the leak only affected surface soil around the identified source area; however, during site characterization activities KAFB learned the leaked fuel had reached the groundwater table and that dissolved-phase fuel contamination migrated northeast and north of KAFB.

2.3 Ongoing Monitoring

2.3.1 Ongoing Soil Vapor Monitoring

Quarterly soil vapor monitoring has been ongoing under the *Vadose Zone Investigation Work Plan* (USACE, 2011) as part of the ST-106/SS-111 investigation to monitor the nature and extent of soil vapor contamination in the vadose zone. A total of 56 soil vapor monitoring locations have been installed during the investigation (Figure 2-1). Each location is comprised of one or more soil vapor monitoring points (SVMPs), for a total of 284 monitoring points. Table 2-1 lists each soil vapor monitoring location, its associated SVMPs, and their associated easting and northing coordinates. The 56 soil vapor monitoring locations include:

- One soil vapor monitoring location contains four SVMPs co-located in the same vault as a groundwater monitoring well in Bullhead Park (KAFB-106028-510; See location number one in Table 2-1 below).

- Thirty-five locations installed in 2010 and 2011, each with six SVMPs, are located throughout the BFF, on base property north of the BFF, on COA property in Bullhead Park and its open space area, and on Veteran's Administration (VA) property. These locations are named using the convention KAFB-106XXX, to signify that they were installed as part of the investigation at SWMU ST-106/SS-111. Numbering at these 35 soil vapor monitoring locations range from KAFB-106108 through KAFB-106142 (See locations two through 36 in Table 2-1 below). Individual SVMPs at each location are further identified using the bottom of the screen depth of each point (e.g. KAFB-106108-050).

- Twenty locations installed inside the BFF referred to with the prefix "soil vapor extraction well (SVEW)-XX" or "soil vapor monitoring well (SVMW)-XX (See locations 37 through 56 in table 2-1 below)."

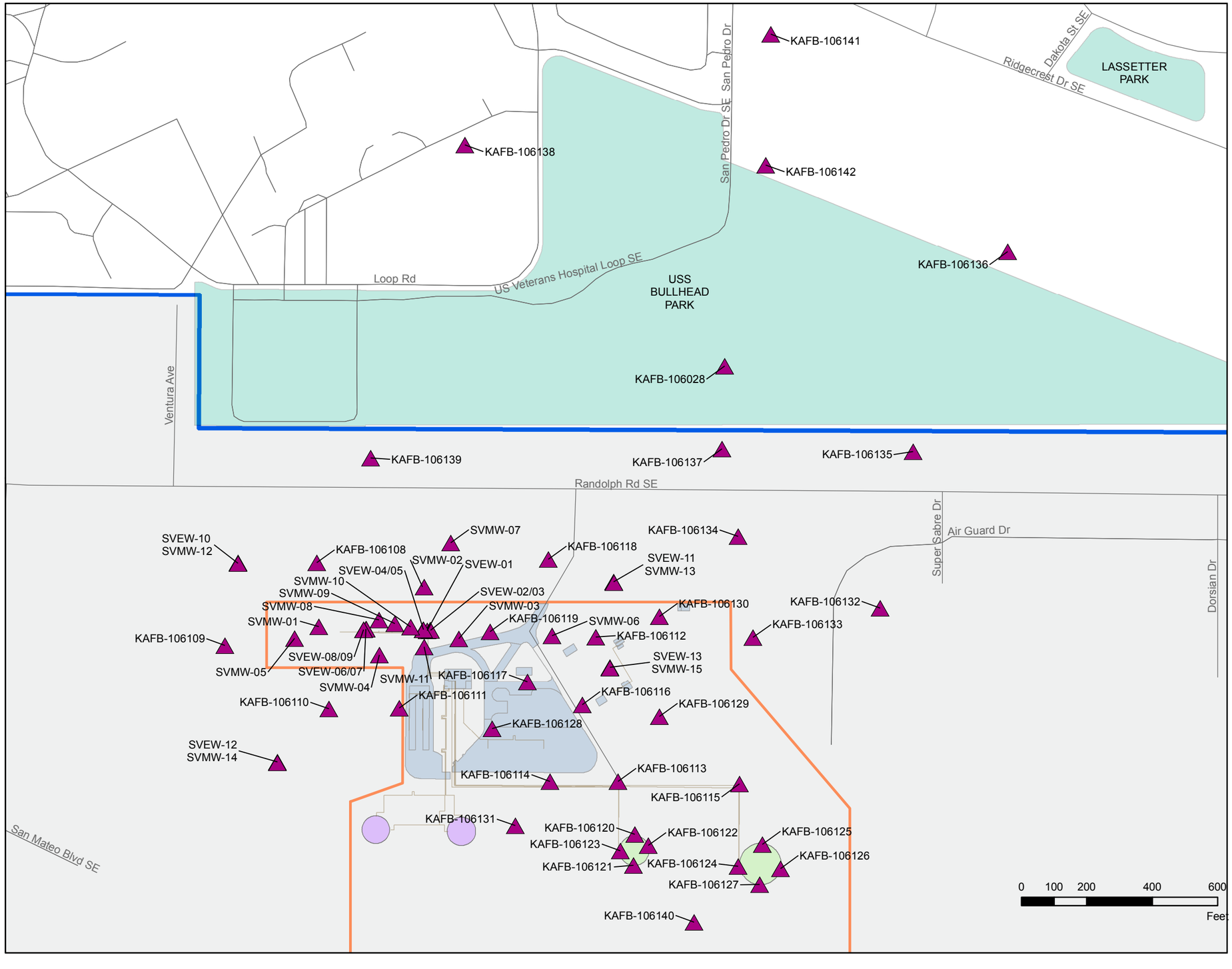
- Four of these 20 locations have both SVMW and SVEW type SVMPs at a single location. [For example, SVMW-13 (comprised of four SVMPs screened between 150 and 450-feet deep) and SVEW-11 (with one SVMP) are located together in a single well vault.

Table 2-1 lists each soil vapor monitoring well location and its associated SVMPs and coordinates.

In first quarter CY 2015, all SVMPs were capped and sealed to minimize barometric-pumping interferences on soil vapor sampling and analyses. Sealing the SVMPs was performed by securing an air-tight cap onto each point/well head and adding a pneumatic quick connect fitting to each monitoring point that serves as a sampling port connection for ease of access and to ensure that an air-tight seal is maintained.

2.3.2 Ongoing Drinking Water Production Well Monitoring

Four drinking water production wells have been sampled monthly as part of the ST-106/SS-111 investigation to confirm that they have not been impacted by groundwater contaminants. These wells include ST106-VA2 on VA hospital property, and KAFB-3, KAFB-15 and KAFB-16 on KAFB property (Figure 2-2). Table 2-2 lists the coordinates of each drinking water well.



Legend

- Kirtland Air Force Base Installation Area
- City of Albuquerque Parks
- Roads
- Bulk Fuels Facility Area
- Soil Vapor Monitoring Location
- Bulk Fuels Facility Infrastructure
- Current Fuel Storage Tanks
- Former Fuel Storage Tanks
- Fuel Transfer Lines

N

Credits: City of Albuquerque

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

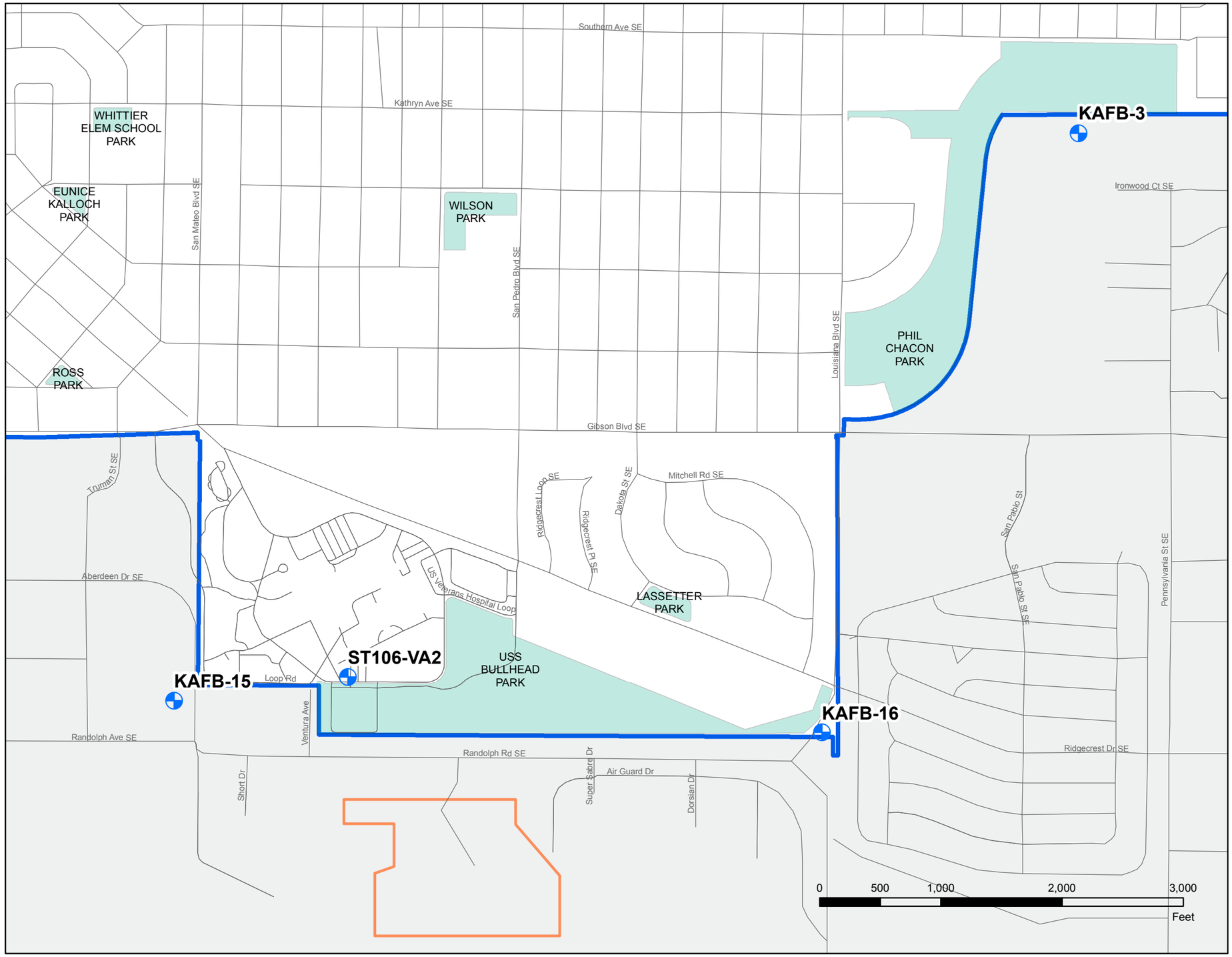
Figure 2-1

Soil Vapor Monitoring Locations
Work Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016



Legend

- Kirtland Air Force Base Installation Area
- City of Albuquerque Parks
- Roads
- Bulk Fuels Facility Area
- Drinking Water Supply Well

N

Credits: City of Albuquerque

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

Figure 2-2

Drinking Water Supply Wells
Work Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
1	KAFB-106028	KAFB-106028-150	401910.441776	1474285.009804
		KAFB-106028-250	401910.491777	1474285.239798
		KAFB-106028-350	401910.751783	1474285.259797
		KAFB-106028-450	401910.851786	1474285.409793
2	KAFB-106108	KAFB-106108-025	400664.903033	1473684.446268
		KAFB-106108-050	400664.773030	1473684.206274
		KAFB-106108-150	400664.993035	1473684.016279
		KAFB-106108-250	400665.243040	1473684.156276
		KAFB-106108-350	400665.173039	1473684.456267
		KAFB-106108-450	400665.023035	1473684.846257
3	KAFB-106109	KAFB-106109-025	400384.736587	1473432.363139
		KAFB-106109-050	400384.916591	1473432.573133
		KAFB-106109-150	400384.736587	1473432.853126
		KAFB-106109-250	400384.456580	1473432.763128
		KAFB-106109-350	400384.446580	1473432.443137
		KAFB-106109-450	400384.676586	1473432.693130
4	KAFB-106110	KAFB-106110-025	400702.613980	1473238.228342
		KAFB-106110-050	400702.373975	1473238.008348
		KAFB-106110-150	400702.523978	1473237.768354
		KAFB-106110-250	400702.783984	1473237.818353
		KAFB-106110-350	400702.833985	1473238.098345
		KAFB-106110-450	400702.033967	1473237.748355
5	KAFB-106111	KAFB-106111-025	400916.998944	1473240.918233
		KAFB-106111-050	400917.178948	1473240.698239
		KAFB-106111-150	400917.438954	1473240.808236
		KAFB-106111-250	400917.418954	1473241.068229
		KAFB-106111-350	400917.118947	1473241.148227
		KAFB-106111-450	400917.038945	1473240.748238
6	KAFB-106112	KAFB-106112-025	401517.592816	1473457.832261
		KAFB-106112-050	401517.752819	1473457.592267
		KAFB-106112-150	401518.032826	1473457.692264
		KAFB-106112-250	401518.062827	1473457.962257
		KAFB-106112-350	401517.772820	1473458.082254
		KAFB-106112-450	401517.052803	1473457.742263
7	KAFB-106113	KAFB-106113-020	401585.424459	1473016.904187
		KAFB-106113-050	401585.284456	1473016.594195
		KAFB-106113-150	401585.534462	1473016.404200
		KAFB-106113-250	401585.844469	1473016.554196
		KAFB-106113-350	401585.754467	1473016.824189
		KAFB-106113-450	401585.554462	1473016.764191

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
8	KAFB-106114	KAFB-106114-025	401377.539646	1473017.104216
		KAFB-106114-050	401377.289640	1473016.884222
		KAFB-106114-150	401377.419643	1473016.594230
		KAFB-106114-250	401377.729650	1473016.614229
		KAFB-106114-350	401377.759651	1473016.924221
		KAFB-106114-450	401377.589647	1473017.374209
9	KAFB-106115	KAFB-106115-025	401956.373050	1473009.614323
		KAFB-106115-050	401956.193046	1473009.374329
		KAFB-106115-150	401955.923040	1473009.414328
		KAFB-106115-250	401955.893039	1473009.694321
		KAFB-106115-350	401956.163045	1473009.754319
		KAFB-106115-450	401956.063043	1473009.754319
10	KAFB-106116	KAFB-106116-025	401475.631878	1473251.167863
		KAFB-106116-050	401475.951886	1473251.187862
		KAFB-106116-150	401475.521876	1473251.417856
		KAFB-106116-250	401475.741881	1473251.627850
		KAFB-106116-350	401476.021887	1473251.467854
		KAFB-106116-450	401475.051865	1473250.747874
11	KAFB-106117	KAFB-106117-025	401308.888006	1473321.085997
		KAFB-106117-050	401309.028009	1473321.305991
		KAFB-106117-150	401308.838004	1473321.555985
		KAFB-106117-250	401308.567998	1473321.445988
		KAFB-106117-350	401308.577998	1473321.165995
		KAFB-106117-450	401308.047986	1473320.746007
12	KAFB-106118	KAFB-106118-025	401372.389414	1473695.475851
		KAFB-106118-050	401372.479416	1473695.765843
		KAFB-106118-160	401372.589419	1473695.285856
		KAFB-106118-265	401372.809424	1473695.735844
		KAFB-106118-350	401372.879425	1473695.385854
		KAFB-106118-450	401372.049406	1473695.735844
13	KAFB-106119	KAFB-106119-025	401194.105322	1473474.311868
		KAFB-106119-050	401194.275326	1473474.061875
		KAFB-106119-150	401194.545332	1473474.151873
		KAFB-106119-250	401194.585333	1473474.421865
		KAFB-106119-350	401194.315327	1473474.531862
		KAFB-106119-450	401194.045321	1473474.741857
14	KAFB-106120	KAFB-106120-025	401636.035658	1472855.058560
		KAFB-106120-050	401636.135660	1472855.248555
		KAFB-106120-150	401636.015658	1472855.498549
		KAFB-106120-250	401635.685650	1472855.268555
		KAFB-106120-350	401635.855654	1472855.038561
		KAFB-106120-450	401635.915655	1472855.388552

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
15	KAFB-106121	KAFB-106121-025	401632.585594	1472760.611118
		KAFB-106121-050	401632.835600	1472760.431123
		KAFB-106121-145	401632.305587	1472760.381125
		KAFB-106121-250	401632.425590	1472760.121132
		KAFB-106121-350	401632.805599	1472760.141131
		KAFB-106121-450	401632.535593	1472760.431123
16	KAFB-106122	KAFB-106122-025	401677.386621	1472821.089473
		KAFB-106122-050	401677.326620	1472821.389465
		KAFB-106122-150	401677.046613	1472821.449464
		KAFB-106122-250	401676.936611	1472821.159472
		KAFB-106122-350	401677.156616	1472820.949477
		KAFB-106122-450	401677.146616	1472821.289468
17	KAFB-106123	KAFB-106123-025	401591.874644	1472805.169919
		KAFB-106123-050	401591.754641	1472805.439911
		KAFB-106123-150	401591.394633	1472805.419912
		KAFB-106123-250	401591.334631	1472805.109920
		KAFB-106123-350	401591.614638	1472804.929925
		KAFB-106123-450	401591.564637	1472805.319915
18	KAFB-106124	KAFB-106124-025	401951.482979	1472757.341154
		KAFB-106124-050	401951.172971	1472757.361154
		KAFB-106124-150	401951.042968	1472757.101161
		KAFB-106124-250	401951.282974	1472756.901166
		KAFB-106124-350	401951.522980	1472757.031163
		KAFB-106124-450	401951.302974	1472757.261156
19	KAFB-106125	KAFB-106125-025	402026.084695	1472824.439325
		KAFB-106125-050	402025.794688	1472824.509323
		KAFB-106125-150	402025.624684	1472824.279330
		KAFB-106125-250	402025.824689	1472824.009337
		KAFB-106125-350	402026.094695	1472824.099334
		KAFB-106125-450	402025.864690	1472824.369327
20	KAFB-106126	KAFB-106126-025	402081.045980	1472750.941306
		KAFB-106126-050	402081.015979	1472751.231298
		KAFB-106126-150	402080.725972	1472751.321296
		KAFB-106126-250	402080.555968	1472751.081302
		KAFB-106126-350	402080.735973	1472750.781310
		KAFB-106126-450	402080.795974	1472751.161300
21	KAFB-106127	KAFB-106127-025	402018.094530	1472701.832646
		KAFB-106127-050	402017.834524	1472701.972642
		KAFB-106127-150	402017.604519	1472701.792647
		KAFB-106127-250	402017.754522	1472701.472656
		KAFB-106127-350	402018.054529	1472701.512655
		KAFB-106127-450	402017.914526	1472701.832646

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
22	KAFB-106128	KAFB-106128-025	401200.655523	1473177.999889
		KAFB-106128-050	401200.545521	1473177.729897
		KAFB-106128-150	401200.765526	1473177.529902
		KAFB-106128-250	401201.025532	1473177.669898
		KAFB-106128-350	401200.965530	1473177.999889
		KAFB-106128-450	401200.045509	1473177.749896
23	KAFB-106129	KAFB-106129-025	401711.597348	1473215.518789
		KAFB-106129-050	401711.877355	1473215.378792
		KAFB-106129-150	401712.097360	1473215.618786
		KAFB-106129-250	401711.977357	1473215.878779
		KAFB-106129-350	401711.687350	1473215.848780
		KAFB-106129-450	401711.817353	1473216.128772
24	KAFB-106130	KAFB-106130-025	401711.947306	1473520.410534
		KAFB-106130-050	401712.157311	1473520.280538
		KAFB-106130-150	401712.427317	1473520.380535
		KAFB-106130-250	401712.377316	1473520.660528
		KAFB-106130-350	401712.107310	1473520.740525
		KAFB-106130-450	401712.057308	1473520.740525
25	KAFB-106131	KAFB-106131-025	401271.447212	1472881.677900
		KAFB-106131-055	401271.667217	1472881.497905
		KAFB-106131-150	401271.567214	1472881.987892
		KAFB-106131-245	401271.937223	1472881.657901
		KAFB-106131-350	401271.837220	1472881.947893
		KAFB-106131-450	401271.047202	1472881.757898
26	KAFB-106132	KAFB-106132-025	402385.372896	1473546.049729
		KAFB-106132-050	402384.882885	1473546.139727
		KAFB-106132-175	402385.202892	1473545.799736
		KAFB-106132-250	402384.892885	1473545.829735
		KAFB-106132-350	402385.152891	1473546.259724
		KAFB-106132-450	402385.072889	1473545.739738
27	KAFB-106133	KAFB-106133-025	401997.193922	1473456.332222
		KAFB-106133-050	401997.113920	1473456.612214
		KAFB-106133-170	401997.353925	1473456.832208
		KAFB-106133-250	401997.593931	1473456.672213
		KAFB-106133-350	401997.543930	1473456.362221
		KAFB-106133-450	401997.063919	1473456.742211
28	KAFB-106134	KAFB-106134-025	401952.322832	1473764.643883
		KAFB-106134-050	401952.042825	1473764.673882
		KAFB-106134-170	401952.432834	1473764.923875
		KAFB-106134-250	401952.232830	1473765.113870
		KAFB-106134-350	401951.932823	1473764.953875
		KAFB-106134-450	401952.282831	1473765.433862

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
29	KAFB-106135	KAFB-106135-025	402486.125151	1474023.666783
		KAFB-106135-050	402486.335156	1474023.456789
		KAFB-106135-150	402486.195152	1474023.146797
		KAFB-106135-250	402485.865145	1474023.256795
		KAFB-106135-350	402485.875145	1474023.556786
		KAFB-106135-450	402486.055149	1474023.526787
30	KAFB-106136	KAFB-106136-025	402775.081742	1474634.810194
		KAFB-106136-050	402775.081742	1474634.910191
		KAFB-106136-150	402775.081742	1474635.010188
		KAFB-106136-250	402775.081742	1474635.110186
		KAFB-106136-350	402775.081742	1474635.210183
		KAFB-106136-450	402775.081742	1474635.310180
31	KAFB-106137	KAFB-106137-025	401902.701639	1474032.086651
		KAFB-106137-050	401902.451633	1474032.276646
		KAFB-106137-150	401902.181627	1474032.086652
		KAFB-106137-250	401902.311630	1474031.816659
		KAFB-106137-350	401902.661638	1474031.816659
		KAFB-106137-450	401902.521634	1474034.116597
32	KAFB-106138	KAFB-106138-025	401117.783308	1474960.381655
		KAFB-106138-050	401117.443300	1474960.271658
		KAFB-106138-150	401117.413299	1474959.981666
		KAFB-106138-250	401117.693306	1474959.871669
		KAFB-106138-350	401117.913311	1474960.121662
		KAFB-106138-450	401117.613304	1474960.211660
33	KAFB-106139	KAFB-106139-025	400829.366788	1474004.577574
		KAFB-106139-050	400829.566792	1474004.137586
		KAFB-106139-150	400829.296786	1474004.027589
		KAFB-106139-250	400829.156783	1474004.327581
		KAFB-106139-350	400829.636794	1474004.477577
		KAFB-106139-450	400829.556792	1474004.807568
34	KAFB-106140	KAFB-106140-025	401817.689909	1472587.305780
		KAFB-106140-050	401818.019916	1472587.175784
		KAFB-106140-150	401817.719909	1472587.635771
		KAFB-106140-250	401818.019916	1472587.695770
		KAFB-106140-350	401818.249922	1472587.445776
		KAFB-106140-450	401817.949915	1472587.515774
35	KAFB-106141	KAFB-106141-025	402051.934886	1475298.122360
		KAFB-106141-050	402052.064889	1475298.382353
		KAFB-106141-170	402051.904885	1475298.602347
		KAFB-106141-250	402051.614878	1475298.502350
		KAFB-106141-350	402051.644879	1475298.222357
		KAFB-106141-450	402051.844884	1475298.452351

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
36	KAFB-106142	KAFB-106142-030	402036.744600	1474898.733172
		KAFB-106142-050	402036.494594	1474898.863168
		KAFB-106142-170	402036.284589	1474898.643174
		KAFB-106142-250	402036.434593	1474898.363182
		KAFB-106142-350	402036.734600	1474898.453179
		KAFB-106142-450	402036.554596	1474898.703173
37	SVEW-01	SVEW-01	401002.570886	1473477.521813
38	SVEW-02/03	SVEW-02/03-060	401012.021105	1473479.611755
		SVEW-02/03-160	401012.091107	1473478.491785
39	SVEW-04/05	SVEW-04/05-313	400990.430605	1473479.771754
		SVEW-04/05-460	400991.320625	1473479.841752
40	SVEW-06/07	SVEW-06/07-060	400815.436552	1473481.961724
		SVEW-06/07-160	400816.096568	1473481.921725
41	SVEW-08/09	SVEW-08/09-260	400807.566370	1473480.611762
		SVEW-08/09-460	400807.936379	1473480.271771
42	SVMW-01	SVMW-01-050	400671.123209	1473489.171553
		SVMW-01-100	400671.443217	1473489.531543
		SVMW-01-250	400671.423216	1473489.171553
		SVMW-01-300	400671.163210	1473489.621541
43	SVMW-02	SVMW-02-050	400993.070644	1473610.618212
		SVMW-02-100	400993.170647	1473610.478215
		SVMW-02-150	400993.220648	1473610.748208
44	SVMW-03	SVMW-03-050	401099.413133	1473452.122485
		SVMW-03-100	401099.553136	1473452.472475
		SVMW-03-250	401099.743141	1473452.272481
		SVMW-03-300	401099.633138	1473451.992488
45	SVMW-04	SVMW-04-050	400855.887502	1473403.243849
		SVMW-04-100	400855.527494	1473403.383845
		SVMW-04-250	400855.527494	1473403.113852
		SVMW-04-300	400855.697498	1473402.813860
46	SVMW-05	SVMW-05-050	400597.061500	1473453.092542
		SVMW-05-100	400596.771493	1473453.362535
		SVMW-05-230	400597.201503	1473453.422533
		SVMW-05-290	400596.971498	1473453.502531
47	SVMW-06	SVMW-06-050	401383.549711	1473462.182165
		SVMW-06-100	401384.009722	1473462.052169
		SVMW-06-252	401383.769716	1473462.402159
		SVMW-06-302	401383.689714	1473461.872174
48	SVMW-07	SVMW-07-050	401074.372504	1473745.824538
		SVMW-07-100	401074.072497	1473745.944535
		SVMW-07-150	401074.272502	1473746.164529

Table 2-1. Soil Vapor Monitoring Point Locations and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded)

Location Number	SVM Location ID	SVMP ID	Easting[1]	Northing
49	SVMW-08	SVMW-08-050	400855.727481	1473509.800964
		SVMW-08-100	400855.777482	1473510.000958
		SVMW-08-250	400855.807482	1473509.770965
50	SVMW-09	SVMW-09-050	400903.768595	1473499.001248
		SVMW-09-100	400903.668593	1473498.981249
		SVMW-09-250	400903.458588	1473499.251241
		SVMW-09-266	400903.188581	1473499.171244
51	SVMW-10	SVMW-10-050	400952.179718	1473488.861515
		SVMW-10-100	400952.159717	1473489.341502
		SVMW-10-150	400952.479724	1473489.071509
		SVMW-10-250	400952.069715	1473488.961512
52	SVMW-11	SVMW-11-050	400992.780668	1473428.233149
		SVMW-11-100	400992.940671	1473428.593139
		SVMW-11-250	400992.410659	1473428.593140
		SVMW-11-260	400992.560663	1473428.813134
53	SVMW-12/ SVEW-10	SVMW-12-150	400424.977477	1473683.186342
		SVMW-12-250	400425.217482	1473683.336338
		SVMW-12-350	400424.887475	1473683.766326
		SVMW-12-450	400424.627469	1473683.536333
		SVEW-10	400424.857474	1473685.086291
54	SVMW-13/ SVEW-11	SVMW-13-150	401572.894069	1473624.437741
		SVMW-13-250	401572.554061	1473624.437741
		SVMW-13-350	401572.634063	1473624.217747
		SVMW-13-450	401572.754066	1473623.977754
		SVEW-11	401572.134051	1473626.047698
55	SVMW-14/ SVEW-12	SVMW-14-150	400544.990358	1473074.172810
		SVMW-14-250	400544.730352	1473074.292806
		SVMW-14-350	400544.780353	1473073.942816
		SVMW-14-450	400545.220363	1473073.932816
		SVEW-12	400545.010358	1473076.332751
56	SVMW-15/ SVEW-13	SVMW-15-150	401560.833833	1473362.634831
		SVMW-15-250	401561.343845	1473362.704829
		SVMW-15-350	401561.013837	1473362.434836
		SVMW-15-450	401561.013837	1473362.994821
		SVEW-13	401560.863833	1473364.984767

[1] Well coordinates are provided in New Mexico State Plane (NAD27).

Table 2-2. Drinking Water Supply Well Names and Coordinates
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Well Location Name	Easting	Northing
ST106-VA2	400544.79	1474576.3
KAFB-3	406572.7	1479061.58
KAFB-15	399113.43	1474381.13
KAFB-16	404456.63	1474118.74

3 TASKS AND ACTIVITIES

This section presents the activities that will be performed under this project. Major divisions include mobilization/demobilization, quarterly soil vapor sampling and analysis, and monthly production well sampling and analysis. This section also describes soil vapor monitoring well installation and abandonment, equipment decontamination, use of personal protective equipment (PPE) and photoionization detector (PID), field quality control and sample packaging and shipping.

3.1 Mobilization/Demobilization

A secure, fenced equipment yard has been established for both equipment and materials for sampling activities both on and off KAFB for this project. A portable office trailer with electrical power has been established inside the secured equipment yard proximal to the BFF at KAFB.

3.2 Soil Vapor Sampling

Sundance will perform quarterly sampling of the existing 284 SVMP network described in section 2.3. All field personnel collecting soil vapor samples are required to be trained and fully understand the sampling procedure outlined in this document. Any and all questions will be addressed prior to the start of sampling via a field sampling orientation led by the Sundance Technical Lead. During the first quarter CY 2016 sampling performed by Sundance, the condition of each well port will be examined by the field personnel to confirm the integrity of each fitting and to immediately address and mitigate any problems or replace any defective parts. The well integrity inspection form can be found in Appendix A, Field Forms.

3.2.1 Pre-Sampling Steps

3.2.1.1 Horiba Model MEXA 584L Calibration

During sampling of each soil vapor well, field parameters including total hydrocarbons (HC), oxygen (O_2), and carbon dioxide (CO_2) will be measured using a Horiba Mexa 584L auto emissions analyzer (Horiba).

The Horiba is sold as an engine exhaust monitoring instrument, and the measurement of field parameters during soil vapor monitoring is not the manufacturer's intended purpose. However, while not the intended use of the instrument, the Horiba's sampling ability and the non-dispersive infrared detector and chemical cell detector make it an appropriate instrument for total soil vapor HC, CO_2, and O_2 analyses.

The Horiba manufacturer's calibration procedure, which was developed for engine exhaust monitoring, has been modified to better calibrate the instrument for measuring soil vapor petroleum HC, O_2 and CO_2 concentrations. The modified calibration method includes a more representative calibration gas, more frequent calibration than specified by the manufacturer, frequent calibration checks (HC, O_2 and CO_2) during daily Horiba usage, and real-time data analysis to look for indicators of potential calibration deviations.

At the beginning of every work day, the Horiba will be calibrated for air-phase petroleum HC and CO_2 against a calibration standard of known concentrations in a premixed gas cylinder. The Horiba will also be calibrated for O_2 against atmospheric concentrations. At the middle of each work day (or no more than 5 hours after the start of work), a calibration check will be performed on the Horiba to determine whether the calibration of any of the parameters has drifted since the morning calibration. If the calibration check results are outside of 5% of the calibration gas standards, then the Horiba will be recalibrated prior to additional sampling.

The same calibration gas cylinder will be used to calibrate every Horiba instrument and the same person will complete calibration at the beginning of each day to ensure consistent calibrations. The calibration gas consists of 1,600 parts per million by volume (ppmv) propane, 13.0% (percent) CO_2, and the remaining volume will consist of nitrogen. This calibration gas mixture was selected to accurately calibrate the Horiba for the gases that are present in the vadose zone during soil vapor sampling and respiration/rebound testing, specifically, CO_2 and HC (calibrated as propane).

The initial daily calibration of the instrument will be performed by applying the pre-mixed gas into the calibration port located on top of the instrument. The calibration steps listed in the Horiba manual for applying the calibration gas and the sequence of key strokes listed will be followed to complete calibration. After calibration, HC and CO_2 concentrations should be within 5% of the known calibration gas values.

The Horiba Instruction Manual goes through the calibration of each compound. During the calibration of HC, the Horiba will display three numbers as shown in the Horiba Instruction Manual. The top number is the known value of the calibration gas (propane at 1,600 ppmv). The middle number will be constant for a particular instrument, but will vary from instrument to instrument; this number is the factory calibration setting for that instrument that corresponds to the input value of the calibration gas. The bottom number will change as the calibration gas is applied and read by the instrument. Both the middle and bottom numbers are reporting the gas as hexane rather than propane; hexane is the standard for HC for the Horiba instrument. The conversion between hexane and propane for the Horiba is approximately one half the concentration. Thus, a reading of 800 ppmv reported as hexane by the detector is the equivalent of 1,600 ppmv as propane. Once the gas is applied, the bottom HC number on the display should read within 5% of the middle number. A value of approximately 800 ppmv HC is expected after the Horiba instrument is calibrated.

After calibration is confirmed, the same calibration gas will be used to fill a 3 liter Tedlar® gas sampling bag. The customized sampling system (Figure 3-1) will be used to complete the calibration as follows:

Step 1. Disconnect all quick connect pneumatic fittings from sampling system with the exception of the hose. This ensures the system is sealed from all points except through the hose.

Step 2. Visually check that the red drain separator O-ring (approximately 2-inches in diameter) is visible at the opening of the sample inlet port, which is located on the front of the Horiba and to the right of the screen display. Insert the male pneumatic fitting on the end of the Horiba sampling tube to the female quick connect on the sampling system and ensure a secure fit.

Step 3. Ensure that the sampling system is purged according to the steps listed below under Section 3.2.1.2 - Cross Contamination Purging for Sampling System.

Step 4. Once within the given values, attach the Tedlar® bag to the male pneumatic fitting at the end of the braided stainless steel tubing.

Step 5. Open the Tedlar® bag valve to allow the calibration gas to be pulled through the sampling system.

Step 6. Record the instrument read-outs when the instrument has stabilized and compare the results to the calibration gas concentrations.

The mid-day calibration check will be performed using the same technique as the "flow though" portion of initial calibration described in steps one through six above.~~. A 3-liter Tedlar bag will be filled with the premixed calibration gas and connected to the customized sample system, which will be connected to the Horiba. The calibration gas will then be pulled through the sample system to the Horiba.~~

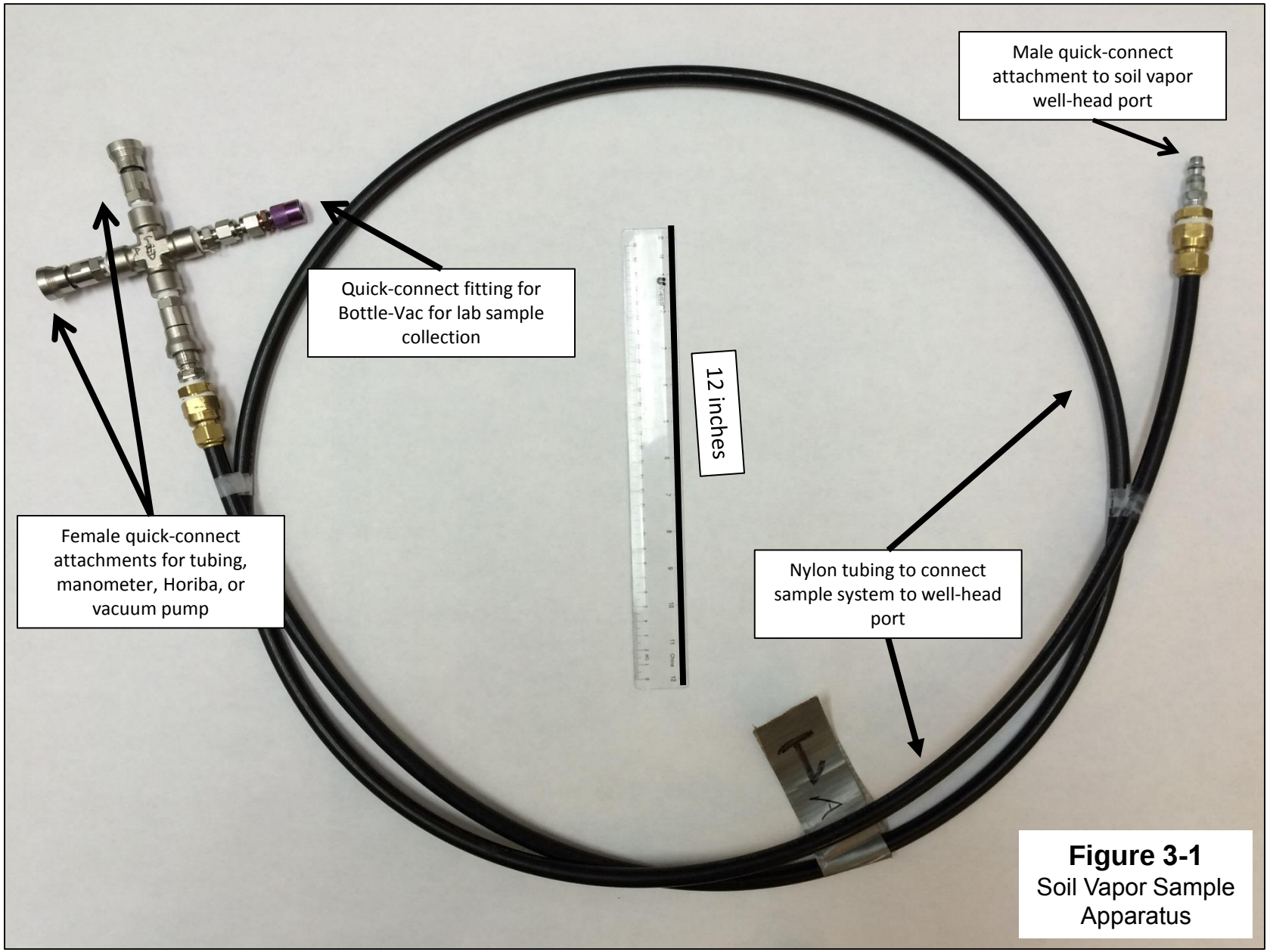


Figure 3-1
Soil Vapor Sample Apparatus

If the values for HC, O_2 and CO_2 are within 5% of the calibration values made using the calibration port, the calibration process is complete. As stated previously, the Horiba read-outs for HC are reported as hexane. A value of approximately 800 ppmv HC is expected. All other gas composition values should match the calibration gas values. If values are outside of this range, perform a leak check as described in Section 3.2.1.3 and follow the calibration process again.

If at any point during sampling, a reading for HC, O_2 or CO_2 reaches an unreasonable value (e.g., an O_2 concentration greater than 22%) or if a data value falls outside the trend indicated by previous readings at a given SVMP, a calibration check will be triggered. The expected range of values are: for HC - from 0 to 40,000 ppmv, for percent O_2 from 0 % to 22%, and for percent CO_2 from 0% to 15%. If any readings are outside of these ranges, a calibration check must be made and if necessary, the instrument will be recalibrated.

3.2.1.2 Cross Contamination Purging for Sampling System

The sampling system must be purged with ambient air before being attached to a SVMP sample port to minimize the potential for cross contamination between sample collections. To ensure the entire sample train is thoroughly purged, attach the pump to the setup and flush atmospheric air through the quick connect port and the nylon tubing. All quick connect pneumatic fittings are to be opened during this process by placing a male fitting into the female fitting to allow for flow. Monitor the purging effectiveness using the Horiba to ensure no contaminants are still present and only ambient air is being read. Correct values for ambient air must be less than 5 ppmv HC, between 20% to 22% for percent O_2, and 0% for percent CO_2. Complete instrument purging must be performed after sampling each SVMP.

3.2.1.3 Leak Check of Sample System

At the beginning of each day, the sampling system will be leak checked by using the pump to apply vacuum to the sampling system as follows:

Step 1. Cap the male pneumatic fitting on the end of the nylon tubing with a spare female quick disconnect fitted to a vacuum/pressure gauge.

Step 2. Connect the SVMP purging/sampling pump to one of the quick disconnect fittings on the sample system and evacuate the air from the sample system to establish a vacuum.

Step 3. Disconnect the pump and immediately record the vacuum reading from the pressure/vacuum gauge.

Step 4. After 10 minutes have elapsed, check and record the vacuum reading on the gauge.

Step 5. Verify that the starting and ending vacuum readings are within 10% to ensure that the sampling system is not leaking.

Step 6. If the two vacuum readings are not within 10% of each other, check the conditions of the seals and repeat the leak test until the sampling system is confirmed to be air tight.

3.2.2 Soil Vapor Sampling Procedures

3.2.2.1 Sample Train Setup

The Horiba analyzer must be turned on, warmed up, and calibrated according to the steps stated above and then attached to the sampling system. The Horiba analyzer is turned on for the first time at the

beginning of the day and remains in the on position throughout the day. The Horiba analyzer is plugged into the 12V DC outlet in the project vehicle using an AC inverter. All other equipment is gas powered or will be powered by generator, and can be powered off between sampling at each well. The pump is attached and sealed to the setup by a quick connect fitting. It is important that no pneumatic fittings besides the tubing to the soil vapor well port are attached prior to turning on the pump.

3.2.2.2 Static Pressure Measurement

Before taking the static pressure reading, the manometer instrument must be zeroed to atmospheric pressure. The screen should read 0.00 inches of water column (in WC). After confirming that the manometer is zeroed, the following procedure is used to connect the sampling system to the SVMPs and measure the static (also called baseline) pressure, to assure readiness for purging and sampling:

Step 1. Connect the manometer to the quick connect on the side of the sampling system opposite of the Bottle-Vac™ sample collection port (see Figure 3-1).

Step 2. Verify that the manometer reads 0.00 in WC.

Step 3. Insert the male quick connect fitting on the end of the nylon tubing to the female quick disconnect fitting on the top of the SVMP and ensure a secure connection.

Step 4. Monitor the change in manometer readings over time and record the pressure/vacuum reading when the meter stabilizes.

Note: Static pressure readings have typically ranged from +2.00 in (pressure) to -12.00 in WC (vacuum) at each soil vapor well port.

3.2.2.3 Well Purging

Stagnant soil vapor is purged from the SVMP as follows:

Step 1. Tum on the SVMP sampling pump, verify the operation of the flow rotameter, and check for potential leaks as necessary.

Step 2. Consult the Purge Table (Table 3-1) for the initial purge volume.

Step 3. Connect the female quick disconnect on the terminal end of the sampling system to the male quick connect on the vacuum side of the soil vapor monitoring sampling pump and start timing the purge cycle. (Note: Use the flow rate on the rotameter and the pre-calculated purge volume to quickly calculate the purge time. The purge time is determined by the well port diameter, well depth, and rate of the pump; all of which are known before sampling with the exception of the flowrate. The amount of vapor needed to be removed is based on one well casing volume.)

Step 4. After adequately purging for the appropriate time, quickly disconnect the sampling system from the vacuum pump. (Note: The sampling system is to remain connected to the SVMP for the duration of sampling.)

Step 5. Allow the manometer reading to return to within 0.10 in. WC of the static pressure reading before moving to the next step in the sampling procedure.

3.2.2.4 Horiba Readings

Once the SVMP has been purged, the following procedure is used to take and record HC, O_2 and CO_2 measurements using the calibrated Horiba:

Step l. Ensure that the Horiba is turned on and functioning properly.

Step 2. Record the manometer reading.

Step 3. Insert the male quick connect fitting into the female quick connect fitting on the terminal side of the sampling system and ensure a tight connection.

Step 4. Observe the Horiba O_2 reading for stability or for a maximum of one minute, whichever comes first.

Step 5. Record the O_2, CO_2, and HC readings and quickly disconnect the Horiba. Photograph the Horiba reading for quality control (QC) reference. Include the well number in the picture.

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
1	KAFB-106028	KAFB-106028-150	148.75	151.25	0.50	0.00136	0.409	0.615
		KAFB-106028-250	248.75	251.25	0.50	0.00136	0.409	0.752
		KAFB-106028-350	348.75	351.25	0.50	0.00136	0.409	0.888
		KAFB-106028-450	448.75	451.25	0.50	0.00136	0.409	1.024
2	KAFB-106108	KAFB-106108-025	15.30	25.30	0.75	0.00307	1.636	1.714
		KAFB-106108-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106108-150	140.20	150.20	0.75	0.00307	1.636	2.097
		KAFB-106108-250	240.30	250.30	0.75	0.00307	1.636	2.404
		KAFB-106108-350	340.30	350.30	0.75	0.00307	1.636	2.711
		KAFB-106108-450	440.00	450.00	3.00	0.04909	1.636	23.725
3	KAFB-106109	KAFB-106109-025	15.20	25.20	0.75	0.00307	1.636	1.713
		KAFB-106109-050	40.10	50.10	0.75	0.00307	1.636	1.790
		KAFB-106109-150	140.00	150.00	0.75	0.00307	1.636	2.096

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106109-250	240.20	250.20	0.75	0.00307	1.636	2.404
		KAFB-106109-350	340.60	350.60	0.75	0.00307	1.636	2.712
		KAFB-106109-450	440.00	450.00	3.00	0.04909	1.636	23.725
4	KAFB-106110	KAFB-106110-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106110-050	40.10	50.10	0.75	0.00307	1.636	1.790
		KAFB-106110-150	140.30	150.30	0.75	0.00307	1.636	2.097
		KAFB-106110-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106110-350	340.20	350.20	0.75	0.00307	1.636	2.710
		KAFB-106110-450	440.00	450.00	3.00	0.04909	1.636	23.725
5	KAFB-106111	KAFB-106111-025	15.20	25.20	0.75	0.00307	1.636	1.713
		KAFB-106111-050	40.10	50.10	0.75	0.00307	1.636	1.790
		KAFB-106111-150	140.30	150.30	0.75	0.00307	1.636	2.097
		KAFB-106111-250	240.30	250.30	0.75	0.00307	1.636	2.404
		KAFB-106111-350	340.40	350.40	0.75	0.00307	1.636	2.711
		KAFB-106111-450	440.30	450.30	3.00	0.04909	1.636	23.740

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
6	KAFB-106112	KAFB-106112-025	15.00	25.00	0.75	0.00307	1.636	1.713
		4KAFB-1506112-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106112-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106112-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106112-350	339.00	349.00	0.75	0.00307	1.636	2.707
		KAFB-106112-450	439.00	449.00	3.00	0.04909	1.636	23.676
7	KAFB-106113	KAFB-106113-020	10.00	20.00	0.75	0.00307	1.636	1.697
		KAFB-106113-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106113-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106113-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106113-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106113-450	440.00	450.00	3.00	0.04909	1.636	23.725
8	KAFB-106114	KAFB-106114-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106114-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106114-150	140.00	150.00	0.75	0.00307	1.636	2.096

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106114-250	235.00	245.00	0.75	0.00307	1.636	2.388
		KAFB-106114-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106114-450	439.60	449.60	3.00	0.04909	1.636	23.706
9	KAFB-106115	KAFB-106115-025	14.60	24.60	0.75	0.00307	1.636	1.711
		KAFB-106115-050	39.60	49.60	0.75	0.00307	1.636	1.788
		KAFB-106115-150	144.60	154.60	0.75	0.00307	1.636	2.110
		KAFB-106115-250	239.60	249.60	0.75	0.00307	1.636	2.402
		KAFB-106115-350	339.60	349.60	0.75	0.00307	1.636	2.709
		KAFB-106115-450	439.60	449.60	3.00	0.04909	1.636	23.706
10	KAFB-106116	KAFB-106116-025	10.00	19.45	0.75	0.00307	1.546	1.606
		KAFB-106116-050	40.00	49.45	0.75	0.00307	1.546	1.698
		KAFB-106116-150	140.00	149.45	0.75	0.00307	1.546	2.005
		KAFB-106116-250	240.00	249.45	0.75	0.00307	1.546	2.311
		KAFB-106116-350	340.00	349.45	0.75	0.00307	1.546	2.618
		KAFB-106116-450	440.00	448.95	3.00	0.04909	1.464	23.502

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
11	KAFB-106117	KAFB-106117-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106117-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106117-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106117-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106117-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106117-450	440.00	450.00	3.00	0.04909	1.636	23.725
12	KAFB-106118	KAFB-106118-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106118-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106118-160	150.00	160.00	0.75	0.00307	1.636	2.127
		KAFB-106118-265	255.00	265.00	0.75	0.00307	1.636	2.449
		KAFB-106118-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106118-450	440.00	450.00	3.00	0.04909	1.636	23.725
13	KAFB-106119	KAFB-106119-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106119-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106119-150	140.00	150.00	0.75	0.00307	1.636	2.096

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106119-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106119-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106119-450	440.00	450.00	3.00	0.04909	1.636	23.725
14	KAFB-106120	KAFB-106120-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106120-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106120-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106120-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106120-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106120-450	434.00	444.00	3.00	0.04909	1.636	23.431
15	KAFB-106121	KAFB-106121-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106121-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106121-145	135.00	145.00	0.75	0.00307	1.636	2.081
		KAFB-106121-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106121-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106121-440	434.00	444.00	3.00	0.04909	1.636	23.431

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
16	KAFB-106122	KAFB-106122-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106122-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106122-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106122-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106122-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106122-450	434.00	444.00	3.00	0.04909	1.636	23.431
17	KAFB-106123	KAFB-106123-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106123-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106123-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106123-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106123-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106123-450	432.00	442.00	3.00	0.04909	1.636	23.333
18	KAFB-106124	KAFB-106124-025	15.10	25.00	0.75	0.00307	1.620	1.696
		KAFB-106124-050	40.10	50.00	0.75	0.00307	1.620	1.773
		KAFB-106124-150	140.10	150.00	0.75	0.00307	1.620	2.080

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106124-250	240.10	250.00	0.75	0.00307	1.620	2.387
		KAFB-106124-350	340.10	350.00	0.75	0.00307	1.620	2.693
		KAFB-106124-450	440.10	450.00	3.00	0.04909	1.620	23.709
19	KAFB-106125	KAFB-106125-025	15.20	25.00	0.75	0.00307	1.603	1.680
		KAFB-106125-050	40.20	50.00	0.75	0.00307	1.603	1.757
		KAFB-106125-150	140.20	150.00	0.75	0.00307	1.603	2.063
		KAFB-106125-250	240.20	250.00	0.75	0.00307	1.603	2.370
		KAFB-106125-350	340.20	350.00	0.75	0.00307	1.603	2.677
		KAFB-106125-450	440.20	450.00	3.00	0.04909	1.603	23.693
20	KAFB-106126	KAFB-106126-025	15.10	25.00	0.75	0.00307	1.620	1.696
		KAFB-106126-050	40.10	50.00	0.75	0.00307	1.620	1.773
		KAFB-106126-150	140.10	150.00	0.75	0.00307	1.620	2.080
		KAFB-106126-250	240.10	250.00	0.75	0.00307	1.620	2.387
		KAFB-106126-350	340.10	350.00	0.75	0.00307	1.620	2.693
		KAFB-106126-450	440.20	450.00	3.00	0.04909	1.603	23.693

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
21	KAFB-106127	KAFB-106127-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106127-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106127-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106127-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106127-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106127-450	440.00	450.00	3.00	0.04909	1.636	23.725
22	KAFB-106128	KAFB-106128-025	15.04	25.04	0.75	0.00307	1.636	1.713
		KAFB-106128-050	40.07	50.07	0.75	0.00307	1.636	1.790
		KAFB-106128-150	140.19	150.19	0.75	0.00307	1.636	2.097
		KAFB-106128-250	240.29	250.29	0.75	0.00307	1.636	2.404
		KAFB-106128-350	340.39	350.39	0.75	0.00307	1.636	2.711
		KAFB-106128-450	440.06	450.06	3.00	0.04909	1.636	23.728
23	KAFB-106129	KAFB-106129-025	15.10	25.10	0.75	0.00307	1.636	1.713
		KAFB-106129-050	39.70	49.70	0.75	0.00307	1.636	1.788
		KAFB-106129-150	140.20	150.20	0.75	0.00307	1.636	2.097

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106129-250	240.10	250.10	0.75	0.00307	1.636	2.403
		KAFB-106129-350	337.40	347.40	0.75	0.00307	1.636	2.702
		KAFB-106129-450	440.70	450.70	3.00	0.04909	1.636	23.760
24	KAFB-106130	KAFB-106130-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106130-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106130-150	150.00	160.00	0.75	0.00307	1.636	2.127
		KAFB-106130-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106130-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106130-450	440.00	450.00	3.00	0.04909	1.636	23.725
25	KAFB-106131	KAFB-106131-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106131-055	45.00	55.00	0.75	0.00307	1.636	1.805
		KAFB-106131-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106131-245	235.00	245.00	0.75	0.00307	1.636	2.388
		KAFB-106131-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106131-450	430.00	440.00	3.00	0.04909	1.636	23.234

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
26	KAFB-106132	KAFB-106132-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106132-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106132-175	164.00	174.00	0.75	0.00307	1.636	2.170
		KAFB-106132-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106132-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106132-450	440.00	450.00	3.00	0.04909	1.636	23.725
27	KAFB-106133	KAFB-106133-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106133-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106133-170	160.00	170.00	0.75	0.00307	1.636	2.158
		KAFB-106133-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106133-350	339.00	349.00	0.75	0.00307	1.636	2.707
		KAFB-106133-450	439.00	449.00	3.00	0.04909	1.636	23.676
28	KAFB-106134	KAFB-106134-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106134-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106134-170	160.00	170.00	0.75	0.00307	1.636	2.158

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106134-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106134-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106134-450	440.00	450.00	3.00	0.04909	1.636	23.725
29	KAFB-106135	KAFB-106135-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106135-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106135-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106135-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106135-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106135-450	440.00	450.00	3.00	0.04909	1.636	23.725
30	KAFB-106136	KAFB-106136-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106136-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106136-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106136-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106136-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106136-450	440.00	450.00	3.00	0.04909	1.636	23.725

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
31	KAFB-106137	KAFB-106137-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106137-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106137-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106137-250	240.10	250.10	0.75	0.00307	1.636	2.403
		KAFB-106137-350	340.50	350.50	0.75	0.00307	1.636	2.711
		KAFB-106137-450	440.00	450.00	3.00	0.04909	1.636	23.725
32	KAFB-106138	KAFB-106138-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106138-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106138-150	140.00	150.00	0.75	0.00307	1.636	2.096
		KAFB-106138-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106138-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106138-450	440.00	450.00	3.00	0.04909	1.636	23.725
33	KAFB-106139	KAFB-106139-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106139-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106139-150	140.00	150.00	0.75	0.00307	1.636	2.096

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		KAFB-106139-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106139-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106139-450	440.00	450.00	3.00	0.04909	1.636	23.725
34	KAFB-106140	KAFB-106140-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106140-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106140-150	141.80	151.80	0.75	0.00307	1.636	2.102
		KAFB-106140-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106140-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106140-450	440.00	450.00	3.00	0.04909	1.636	23.725
35	KAFB-106141	KAFB-106141-025	15.00	25.00	0.75	0.00307	1.636	1.713
		KAFB-106141-050	50.00	60.00	0.75	0.00307	1.636	1.820
		KAFB-106141-170	160.00	170.00	0.75	0.00307	1.636	2.158
		KAFB-106141-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106141-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106141-450	440.00	450.00	3.00	0.04909	1.636	23.725

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
36	KAFB-106142	KAFB-106142-030	20.00	30.00	0.75	0.00307	1.636	1.728
		KAFB-106142-050	40.00	50.00	0.75	0.00307	1.636	1.789
		KAFB-106142-170	160.00	170.00	0.75	0.00307	1.636	2.158
		KAFB-106142-250	240.00	250.00	0.75	0.00307	1.636	2.403
		KAFB-106142-350	340.00	350.00	0.75	0.00307	1.636	2.710
		KAFB-106142-450	440.00	450.00	3.00	0.04909	1.636	23.725
37	SVEW-01	SVEW-01-260	245.00	260.00	2.00	0.02182	2.454	8.126
38	SVEW-02/03	SVEW-02-060	45.00	60.00	2.00	0.02182	2.454	3.763
		SVEW-03-160	145.00	160.00	2.00	0.02182	2.454	5.945
39	SVEW-04/05	SVEW-04-313	298.00	313.00	2.00	0.02182	2.454	9.283
		SVEW-05-460	445.00	460.00	2.00	0.02182	2.454	12.490
40	SVEW-06/07	SVEW-06-060	45.00	60.00	2.00	0.02182	2.454	3.763
		SVEW-07-160	145.00	160.00	2.00	0.02182	2.454	5.945
41	SVEW-08/09	SVEW-08-260	245.00	260.00	2.00	0.02182	2.454	8.126
		SVEW-09-460	443.00	457.00	2.00	0.02182	2.290	12.261
42	SVMW-01	SVMW-01-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-01-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-01-250	250.70	253.20	0.50	0.00136	0.409	0.754
		SVMW-01-300	308.50	310.00	0.50	0.00136	0.245	0.668
43	SVMW-02	SVMW-02-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-02-100	97.00	99.50	0.50	0.00136	0.409	0.545
		SVMW-02-150	150.00	152.50	0.50	0.00136	0.409	0.617
44	SVMW-03	SVMW-03-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-03-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-03-250	250.00	252.50	0.50	0.00136	0.409	0.753

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
		SVMW-03-300	300.00	302.50	0.50	0.00136	0.409	0.821
45	SVMW-04	SVMW-04-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-04-100	98.00	100.50	0.50	0.00136	0.409	0.546
		SVMW-04-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-04-300	297.50	300.00	0.50	0.00136	0.409	0.818
46	SVMW-05	SVMW-05-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-05-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-05-230	229.50	231.00	0.50	0.00136	0.245	0.560
		SVMW-05-290	287.50	290.00	0.50	0.00136	0.409	0.804
47	SVMW-06	SVMW-06-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-06-100	99.50	102.00	0.50	0.00136	0.409	0.548
		SVMW-06-252	252.00	254.50	0.50	0.00136	0.409	0.756
		SVMW-06-302	302.50	305.00	0.50	0.00136	0.409	0.825
48	SVMW-07	SVMW-07-050	49.50	52.00	0.50	0.00136	0.409	0.480
		SVMW-07-100	95.50	98.00	0.50	0.00136	0.409	0.543
		SVMW-07-150	147.50	150.00	0.50	0.00136	0.409	0.614
49	SVMW-08[2]	SVMW-08-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-08-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-08-250	250.00	252.50	0.50	0.00136	0.409	0.753
50	SVMW-09	SVMW-09-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-09-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-09-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-09-266	266.00	268.50	0.50	0.00136	0.409	0.775
51	SVMW-10	SVMW-10-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-10-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-10-150	150.00	152.50	0.50	0.00136	0.409	0.617
		SVMW-10-250	250.00	252.50	0.50	0.00136	0.409	0.753

Table 3-1. Soil Vapor Monitoring Point Construction Parameters and Pre-Calculated Purge Volumes
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded)

Location Number	SVM Location ID	SVMP ID	Screened Interval (ft)		Well Diameter (in)	Casing Area (sq ft)	Filter Pack Volume (cu ft)[1]	Purge Volume (cu ft)
			Top	Bottom				
52	SVMW-11	SVMW-11-050	50.00	52.50	0.50	0.00136	0.409	0.481
		SVMW-11-100	100.00	102.50	0.50	0.00136	0.409	0.549
		SVMW-11-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-11-260	260.00	262.50	0.50	0.00136	0.409	0.767
53	SVMW-12/ SVEW-10	SVMW-12-150	150.00	152.50	0.50	0.00136	0.409	0.617
		SVMW-12-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-12-350	350.00	352.50	0.50	0.00136	0.409	0.890
		SVMW-12-450	450.00	452.50	0.50	0.00136	0.409	1.026
		SVEW-10-410	400.00	410.00	2.00	0.02182	1.636	10.581
54	SVMW-13/ SVEW-11	SVMW-13-150	150.00	152.50	0.50	0.00136	0.409	0.617
		SVMW-13-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-13-350	350.00	352.50	0.50	0.00136	0.409	0.890
		SVMW-13-450	450.00	452.50	0.50	0.00136	0.409	1.026
		SVEW-11-410	400.00	410.00	2.00	0.02182	1.636	10.581
55	SVMW-14/ SVEW-12	SVMW-14-150	150.00	152.50	0.50	0.00136	0.409	0.617
		SVMW-14-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-14-350	350.00	352.50	0.50	0.00136	0.409	0.890
		SVMW-14-450	450.00	452.50	0.50	0.00136	0.409	1.026
		SVEW-12-410	400.00	410.00	2.00	0.02182	1.636	10.581
56	SVMW-15/ SVEW-13	SVMW-15-150	150.00	152.50	0.50	0.00136	0.409	0.617
		SVMW-15-250	250.00	252.50	0.50	0.00136	0.409	0.753
		SVMW-15-350	350.00	352.50	0.50	0.00136	0.409	0.890
		SVMW-15-450	450.00	452.50	0.50	0.00136	0.409	1.026
		SVEW-13-410	400.00	410.00	2.00	0.02182	1.636	10.581

[1]borehole casing factor = (((10 inch diameter/12 inches)^2)(3.14159/4))(0.3 porosity)= 0.1636

[2]SVMW-08-266 is clogged and cannot be sampled

3.2.2.5 Bottle-Vac™ Sampling

The following procedure will be used when collecting Bottle-Vac™ samples for laboratory analyses:

Step 1. Check the vacuum in a Bottle-Vac™ prior to sampling by taking a vacuum reading using a lab-supplied vacuum gauge. The gauge is connected to the bottle through a quick connect set in the same way that the bottle is connected to the sampling system. (Note: Bottles are required to be within 10% of -26 inches of mercury (in Hg) of vacuum. If a lower vacuum is measured, do not use the Bottle-Vac™ for sample collection.)

Step 2. With the nylon tubing still connected to the well port, record the static pressure indicated on the manometer read-out.

Step 3. Connect the Bottle-Vac™ to the specialized female pneumatic connection on the sample system (see Figure 3-1).

Step 4. Disconnect the Bottle-Vac™ after two minutes or once the manometer reading returns to static pressure, whichever comes first.

Step 5. Check the vacuum in the Bottle-Vac™ after removing from the sampling system. (Note: The vacuum should read no higher than 0.10 in Hg of vacuum. If there is still a positive vacuum reading, re-attach the Bottle- Vac™ to the sampling system for two additional minutes before checking the pressure once more; repeat as necessary.)

Bottle-Vacs will be shipped weekly to ALS Environmental Laboratories (ALS) in Simi Valley California, where they will be analyzed for the following analytical methods:

- Volatile organic compounds (VOC) in air by modified method TO-15
- Ethylene dibromide (EDB) by California Air Resources Board method 422
- Air-Phase Petroleum Hydrocarbons (APH) by MA APH 1.0
- Fixed Gases (H_2, Carbon Monoxide, CO_2, N_2, CH_4 and O_2/Ar) by modified U.S. Environmental Protection Agency (EPA) Method 3C

3.2.2.6 Humidity Measurements

Field measurements of subsurface humidity may be collected during future soil vapor sampling events to evaluate the water activity potential in the vadose zone for the assessment of natural biodegradation conditions.

A humidity probe will be used to collect soil-gas humidity and temperature measurements following SVMP purging and sample collection. The humidity probe will be calibrated each day it is used following the manufacturer instructions. The probe will be kept at a steady temperature while being calibrated.

Once calibrated, the humidity probe will be connected to the SVM point. The probe end of the instrument will be sealed so that only soil-gas humidity is read. Once connected to the SVM point, the vacuum pump will be connected to the top of the flow-through chamber so that soil-gas is pulled past the probe. The instrument will not read as accurately in non-moving air. Both the temperature and relative humidity of the soil-gas will be recorded.

3.2.3 Annual Monitoring Network Maintenance

Operations and maintenance for the soil vapor monitoring network will be performed during the sampling events. Wellheads will be inspected for integrity and necessary repairs will be performed as soon as possible. The findings of the inspections and the repairs will be documented by photographs and on the appropriate field forms (Appendix A).

3.3 Drinking Water Production Well Sampling

Sundance will perform monthly sampling of four drinking water production wells: KAFB-3, KAFB-15, KAFB-16 and VA-2 (Figure 2-2). These existing drinking water production wells at KAFB and the VA Hospital actively provide drinking water to the facilities' employees and inhabitants. Because the wells will be actively producing water during sampling, water levels at these wells will not be measured prior to sampling. In addition, one well volume will not be purged prior to sampling. Sampling at the drinking water production wells will be performed in accordance with the following steps:

Step 1. A Yellow Springs Instruments (YSI) 556 multi-probe system multi-parameter instrument will be used to collect field readings for dissolved oxygen (DO), pH, oxidation reduction potential (ORP), conductivity, and temperature during sampling. Calibrate the YSI according to the manufacturer's instructions for pH, ORP, conductivity and DO. Record the readings in a calibration log. Turbidity will be measured using a portable turbidimeter.

Step 2. Decontamination of the YSI will take place before use at each drinking water supply well location. Decontamination will entail rinsing the YSI to remove contaminants of concern that may impact study objectives. Specifications for decontamination materials are as follows:

- Use a standard brand of phosphate-free laboratory detergent, preferably either liquid Liquinox® or powder Alconox®.
- Use bottled water for the wash. Soap and water will remove the gross contamination from the sampling equipment.
- Use deionized water for the final rinse of YSI probes and containers that have direct contact to the sampling medium.

Step 3. Place a bucket underneath the sample port at the wellhead, and open the sample port. Purge any water in the sample port for thirty seconds to ensure that any accumulated sediment is removed.

Step 4. Fill the lower container of the YSI from the sample port and take a baseline reading of DO, pH, ORP, conductivity, and temperature. Fill the sample cell of the portable turbidimeter and collect the turbidity reading. Record these parameters on the sample collection log.

Step 5. Fill the water sample containers in accordance with requirements of the QAPjP (Appendix C). Samples for volatile organic analysis will be collected first. The sample bottles will be carefully filled to avoid overflow and potential loss of preservative, and tapped so entrapment of air is minimized and no head space exists. If bubbles appear, the vial will be refilled or a new vial will be used if a sample preservative (e.g., hydrochloric acid) is used.

Step 6. Place analytical samples in a cooler and chill to 4 degrees Celsius (°C). Samples must be shipped to the appropriate laboratory within 24 hours. The sample cooler must be shaded from direct sunlight immediately after collection.

Step 7. The field logbook, sample log sheet, labels, custody seals, and chain-of-custody forms will be filled out during sample collection.

Drinking water production well samples will be shipped to ALS laboratory in Kelso, WA where they will be analyzed for the following analytical parameters:

- EDB by EPA Method 504.1
- Benzene, toluene, ethylbenzene, and xylenes (BTEX) by EPA Method 524.2

3.4 Soil Vapor Monitoring Well Abandonment

Soil vapor monitoring location and well abandonment techniques and details will be provided in future revisions of this WP. Once the monitoring location(s) to be abandoned has been determined, the specifications will be submitted describing requirements for abandonment.

3.5 Soil Vapor Monitoring Well Installation

Drilling techniques and details on the design and installation of the soil vapor monitoring location(s) will be provided in future revisions of this WP. Once the design of the SVMPs has been completed, then specifications will be submitted describing the installation requirements for these locations.

3.6 Equipment Decontamination

The objective of field decontamination is to remove contaminants of concerns from sampling, and other field equipment to concentrations that will not impact study objectives.

Decontamination procedures specific to soil vapor sampling are outlined in Section 3.2.1.2. It is not anticipated that any additional decontamination procedures will be required for soil vapor sampling.

Decontamination procedures specific to drinking water supply well sampling are outlined in Step 2 of Section 3.3. It is not anticipated that any additional decontamination procedures will be required for drinking water supply well sampling.

3.7 Personal Protective Equipment

Modified Level D PPE will be worn during sampling as described in the project APP, which was submitted under a separate cover. Please reference Section 9.0 of the APP for more detailed information on PPE.

3.8 Photoionization Detector

A PID will be used for breathing zone monitoring during sampling activities. The PID will be calibrated and tested as required in the QAPjP (Appendix C).

3.9 Field Quality Control

Field QC samples will be collected throughout field investigation activities to ensure the integrity and reproducibility of data. Field QC samples include duplicates and trip blanks for VOC analysis.

Field QC samples are discussed in the QAPjP (Appendix C) and are listed below:

- Field duplicate samples (water/vapor) – 10% of total number of environmental samples per event
- Matrix spike/matrix spike duplicate samples (water) – 5% of total number of environmental samples per event
- Trip blank samples (water/vapor) – one per each shipment of groundwater and vapor samples per event for VOCs only
- Temperature blank (water) – 1 per each shipment of environmental samples

3.10 Sample Packaging and Shipping

The primary objective of sample packaging and shipping requirements is to maintain sample integrity from the time a sample is collected until it is received at the analytical laboratory. Chain-of-custody forms, sample labels, custody seals, and other sample documents will be completed as specified in the QAPjP, provided in Appendix C. Specific procedures for packaging and shipping of environmental samples are presented below:

Step 1. A sample label is attached to the sample bottle and completed with indelible ink.

Step 2. For water samples, a cooler (such as a Coleman or other sturdy cooler) will be used as a shipping container. In preparation for shipping samples, the drain plug will be taped shut so that no fluids, such as melted ice, will drain out of the cooler during shipment. A large plastic bag may be used as a liner for the cooler and packing material, such as bubble wrap, or Styrofoam beads, will be placed in the bottom of the liner. All water samples for chemical analysis must be shipped cooled to 4 °C with ice. All samples will require icing prior to shipping.

Step 3. Soil vapor samples will be returned to the lab in the sample container boxes in which they were sent. There are no temperature or preservative requirements for shipping of the soil vapor samples.

Step 4. The liner will be taped closed, if used, and sufficient packing material will be used to prevent sample containers from making contact or rolling around during shipment.

Step 5. A copy of the completed chain-of-custody record will be placed inside the cooler or box.

Step 6. The cooler or box will be closed and taped shut with packing tape.

Step 7. Custody seals will be placed on the cooler or box.

Step 8. The cooler or box will be shipped in accordance with the particular sample media and corresponding hold times.

3.11 Investigation-Derived Waste

It is not anticipated that any investigation-derived waste (IDW) will be generated during soil vapor sampling. ~~Soil vapor sampling does not generate any containerized waste~~.

In addition, all groundwater generated during drinking water supply well sampling events will be 100% captured and contained. Fluids purged or generated at the wellheads will be placed, to the acceptable filling capacity, in 5-gallon buckets that are secured to a truck bed and upon conclusion of the work day, will be discharged to the GWTS through the sump in the building floor. Prior to discharge to the GWTS, sample documentation will be provided to the GWTS operator demonstrating that the water to be discharged has two consecutive preceding sampling events documenting no contaminants meet the definitions of characteristic hazardous waste (40 Code of Federal Regulations [CFR] Part 261). If any previous sampling data are reported above the concentrations stated in 40 CFR Part 261, the IDW will be stored in a 90-day storage facility where the drums will be labeled and stored pending laboratory analytical results. All water investigation-derived waste (IDW) and decontamination water from equipment cleaning across all site activities will be considered non-hazardous water.

The storage containers will not be left unattended. The quantity of water purged from each well and the total quantity of water transferred to the GWTS will be recorded. A minimal amount of fines are anticipated to be present in this water and pre-filtering before batching into the GWTS is not anticipated. If for any reason the GWTS cannot accept the purge water as it is generated (e.g., shut down for maintenance), the water will be temporarily stored in the IDW area on pallets and properly labeled until it can be discharged to the GWTS sump.

Non-reusable PPE will be disposed of in accordance with the project APP (Section 9.0). Any additional waste associated with sampling (plastic bags, paper waste, etc.) will be collected and disposed of via the COA waste management system.

IDW management details pertaining to any well installation or abandonment that may be performed will be provided in Section 7 of future revisions of this WP. Once the drilling techniques and waste streams are determined, specifications will be submitted describing the management of that waste.

3.12 Reporting

Analytical data collected during soil vapor and drinking water monitoring activities described in this WP will be included in the Periodic Monitoring Reports delivered to NMED (quarterly, or the most current NMED-approved reporting requirement) as part of the investigation at SWMU ST-106/SS-111.

First, second and third quarter CY reports will document the monitoring activities performed during each quarter and will provide the detailed information listed below. The fourth quarter CY report may have additional requirements or information, and include cumulative information from the entire year.

- Descriptions of field activities performed during the quarter
- Tables of analytical concentrations
- Maps illustrating contaminant concentrations at specific well locations
- Analytical laboratory data
- Data validation summary of laboratory data and discussion of data quality

4 PROJECT SCHEDULE

The project schedule is provided in Appendix B of this WP.

5 ORGANIZATIONAL PLAN

The organizational structure of the Sundance Team is shown on Figure 5-1. Table 5-1 summarizes the responsibilities, qualifications, and authorities of project team members.

Figure 5.1. Project Team Organization
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

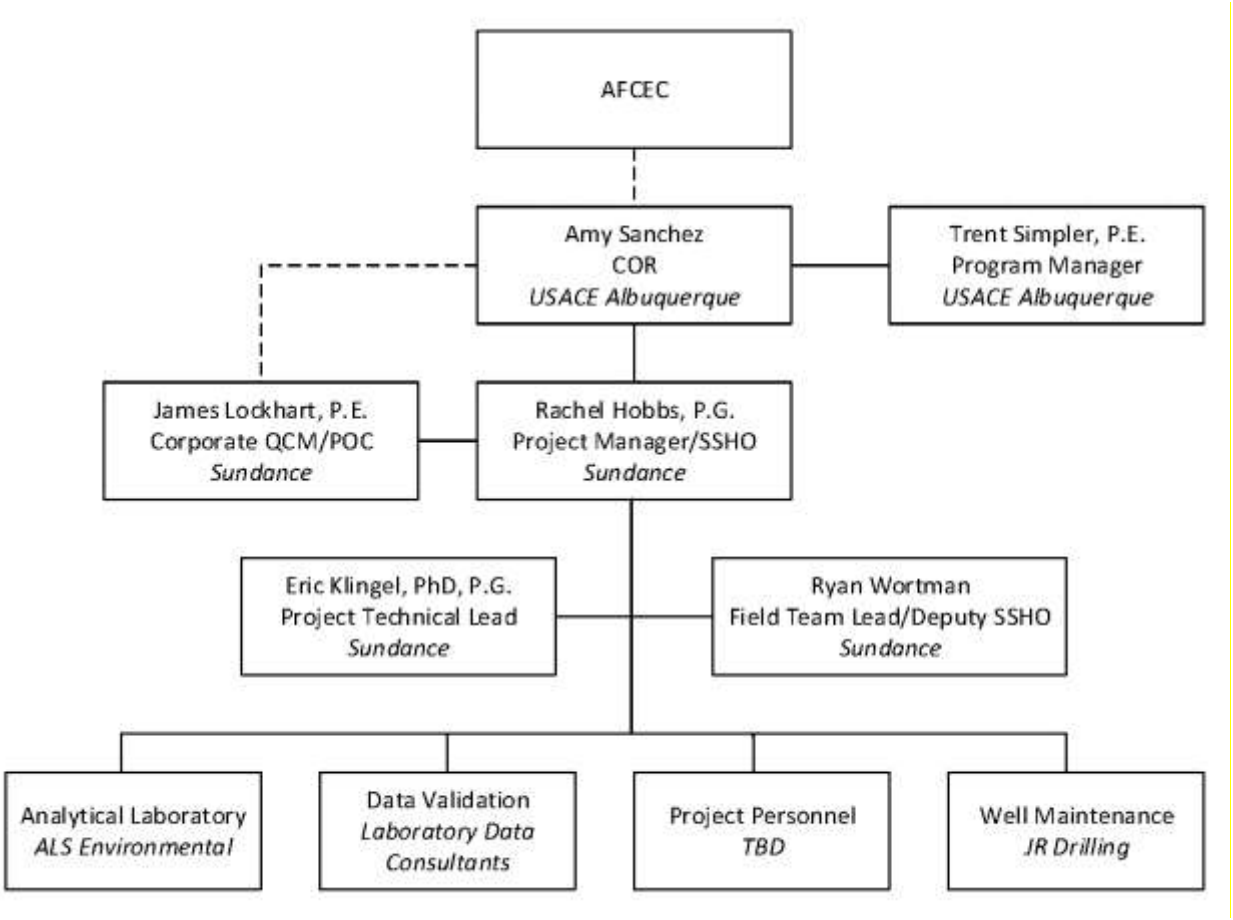


Table 5-1. Staff Roles, Responsibilities, and Authorities
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Position/Staff	Qualifications	Responsibilities	Authority Level
Vice President of Operations Jim Lockhart, P.E.	• BSME, MBA; • 33 years' experience in environmental remediation and engineering; • 25 years in management of environmental and engineering projects.	• As a Sundance Officer, authorized to negotiate and commit resources; • Primary point-of-contact for USACE on contractual and programmatic items; • Ensures consistency in deliverables and cost/performance reporting and progress reporting/invoicing; • Coordinates issue resolution as needed with Contracting Officer's Representative and/or Contracting Officer.	• Coordinates corrective action at programmatic level.
~~Project Manager~~ ~~Patrick Scher, P.G.~~ Project Manager/Site Safety and Health Officer Rachel Hobbs, P.G.	~~• M.S in Geology;~~ ~~• Registered Professional Geologist in two states;~~ ~~• 30 years' experience in environmental remediation/compliance;~~ ~~• 25 years' experience in DoD Project and Program Management;~~ ~~• 15 years' experience as technical lead/project manager on complex environmental projects;~~ ~~• 30-year accident free track record.~~ • M.S. in Geology; • Registered Professional Geologist in the state of Tennessee; • 5 years' experience in environmental remediation; • Past experience coordinating Kirtland BFF project tasks.	• Ensures that all work is accomplished with adequate internal controls; • Main point of contact for USACE on project-specific matters; • Reviews/confirms technical approach from kickoff meeting and throughout project execution to ensure project objectives are met; • Assembles and schedules resources; • Ensures on-schedule and high-quality services are delivered within budget; • Manages subcontractors;	• Full responsibility and authority to execute Task Orders; • Approves subcontractors' invoices, project charges, and deliverables; • Implements corrective action; • Stops work for any reason related to the project. • Approves APPs/SSHPs and all modifications before issuance to USACE; • Manages Health and Safety Program and directs training and required attendance;

Table 5-1. Staff Roles, Responsibilities, and Authorities
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued)

Position/Staff	Qualifications	Responsibilities	Authority Level
		• Coordinates Sundance's participation in the Identifies and mitigates risks related to execution of the technical aspects of the work and ensures site safety; • Ensures work is performed in accordance with USACE/U.S. Air Force Guidelines, state/federal regulations; • Applies lessons learned from current and past projects; • Responsible for front and back end transition activities to ensure continuity on the project; • Ensures public relations sensitivities are met.	• Investigates safety concerns raised by staff; • Investigates any accidents;
~~Project Technical Lead/Site Safety and Health Officer~~ ~~Rachel Hobbs, P.G.~~ Project Technical Lead Eric Klingel, PhD, P.G.	~~• M.S. in Geology;~~ ~~• Registered Professional Geologist in the state of Tennessee;~~ ~~• 5 years' experience in environmental remediation;~~ ~~• Past experience coordinating Kirtland BFF project tasks.~~ • PhD. in Geology; • Registered Professional Geologist in the state of North Carolina and South Carolina; • 30 years' experience in environmental site characterization, remediation and project management.	• Reports to the Project Manager and serves as the Alternative Project Manager; • Overall responsibility for design, implementation, and management of sampling activities; • Reviews all work plans, reporting, and data deliverables; • Coordinates with Field Personnel for oversight and QC; • Responsible for providing input for the design of the corrective actions	~~• Approves APPs/SSHPs and all modifications before issuance to USACE;~~ ~~• Manages Health and Safety Program and directs training and required attendance;~~ ~~• Investigates safety concerns raised by staff;~~ ~~• Investigates any accidents;~~ • Stops work for any reason including noncompliance /safety violation, or quality violations.

Table 5-1. Staff Roles, Responsibilities, and Authorities
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued

Position/Staff	Qualifications	Responsibilities	Authority Level
		and reviews corrective elements specific to sampling; • Oversees development of APP in accordance with Engineer Manual 385-1-1 and Occupational Safety and Health Administration regulations; • Assists Project Manager and procurement staff in verification of safety performance of subcontractors Investigates any incidents, accidents, or safety violations Performs safety audits; • Manages monitoring reports.	
Field Team Lead/Deputy Site Safety and Health Officer Ryan Wortman	• B.S. in Geology; • Past experience coordinating Kirtland BFF project tasks.	• Reports to Technical Lead and/or Project Manager; • Oversees sampling team and sampling activities; • Coordinates with the Project Manager and Project Technical Lead on any deviations from the QAPjP due to changed field conditions such that data quality objectives are met; • Coordinates with SSHO to ensure that project activities are being performed in accordance with the APP. • Performs Health and Safety oversite in SSHO's absence	• Investigates safety concerns raised by staff; • Stop sampling work at any time due to safety or quality violations.

BFF – Bulk Fuels Facility
B.S. – Bachelor of Science Degree
M.S. – Master of Science Degree
P.E. – Professional Engineer
P.G. – Professional Geologist
QAPjP – Quality Assurance Project Plan
SSHO – Site Safety and Health Officer
SSHP – Site Safety and Health Plan
USACE – US Army Corps of Engineers

6 DATA MANAGEMENT PLAN

Environmental laboratory services will be provided only by laboratories compliant with the *DoD Quality Systems Manual for Environmental Laboratories, Version 5.0* (DoD, 2013) or a most recent version and that hold a current DoD Environmental Laboratory Accreditation Program accreditation for all appropriate analytical methods (DoD, 2013). ALS will provide analytical results in support of this project. ALS will provide electronic data in the Environmental Resource Program Information Management System (ERPIMS) format. The ERPIMS deliverable will be validated for upload to the U.S. Air Force (Air Force) data repository. All analytical data generated in support of this project will be uploaded to the Air Force Data Repository.

Analytical data generated in support of this project will undergo an EPA level III data review by Laboratory Data Consultants, Inc. (LDC). Automated data review software, developed by LDC, will be used to perform 100% EPA Level III data review. The data review will be performed for the monthly drinking water supply well data, as well as soil vapor analytical data obtained from each of the quarterly monitoring events. The data review will be performed using the QC criteria specified in Section 4.0 of the project QAPjP (Appendix C).

ERPIMS Version 5.0 submittals will be reviewed for accuracy and completeness before submittal. ERPIMS submittals will be provided to the Air Force, at a minimum, every six months or as appropriate for data generation for uploading to the Air Force data repository. Submittals will be deemed complete upon receipt of the insertion letter from the Air Force.

All project-related data will be maintained and archived in the electronic project files on the corporate server and will be made available to the government as necessary. All data generated in support of this contract will be maintained in accordance with the contract requirements.

7 INVESTIGATION-DERIVED WASTE MANAGEMENT PLAN

Additional project activities that require an IDW management plan may be required as part of this project. If any such project activities are preformed, an IDW management plan will be included in this section as part of a revision to this WP.

8 REFERENCES

DoD. 2013. *DoD Quality Systems Manual for Environmental Laboratories, Version 5.0.* July.

New Mexico Environment Department (NMED). 2010. Hazardous Waste Treatment Facility Operating Permit, EPA ID No. NM9570024423, issued to U.S. Air Force for the Open Detonation Unit Located at Kirtland Air Force Base, Bernalillo County, New Mexico, by the NMED Hazardous Waste Bureau. July.

Sundance Consulting, 2016. *Quarterly Soil Vapor Sampling and Monthly Drinking Water Sampling Accident Prevention Plan*. Sundance Consulting, Inc., February 2016.

USACE, 2011. *Vadose Zone Investigation Work Plan Bulk Fuels Facility Spill Solid Waste Management Units ST-106 and SS-111.* Prepared by Shaw Environmental and Infrastructure, Kirtland Air Force Base, New Mexico. March.

Appendix A
Field Forms

Field Activity Log

Job Number:	Task Description:	Date:

Weather:	Important Notes:	Sundance Employes Onsite:	Visitors:

Name:	Signature:	Date:

Field Activity Log (Continuation)

Page ____ of _____

Job Number:	Task Description:	Date:

Sundance Consulting Inc.

Well Integrity Checklist

Well ID: _____ **Inspector's Name:** _____
Date: _____
Time: _____ **Inspector's Signature:**_____

Before Opening Well

1. Is well cement pad in good condition? _____
2. Is lid securely tightened to vault?_____
3. Is well clearly labeled? _____
4. Do wells outside of BFF have security bolts? _____
5. Photograph well.

After Removing Lid Before Sampling Well

1. Is gasket worn or damaged? _____
2. Is vault flooded? _____
3. Are ports capped/labeled? _____
4. Are ports angled correctly? _____
5. Are all fittings and quick connects intact and operational?

   _____
6. Can you hear well breathing? _____
7. Photograph well with lid off.

During Sampling

1. Do all quick connects fit securely to sample system? _____
2. Does static pressure after purging return to initial static pressure within one minute?

   _____
3. Is well clogged? _____

Comments:_____

_____

Horiba Calibration Sheet

Horiba ID #: _____________

	Know Gas Mixture			Initial Horiba Reading			Post Cal. Horiba Readings			Flow Through Readings			
Date/Time	C3H8(ppm)	% CO2	% O2*	HC(ppm)	% CO2	% O2*	HC(ppm)	% CO2	% O2*	HC(ppm)	% CO2	% O2*	Initals
	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										
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	1600	13	20.9										
	1600	13	20.9										
	1600	13	20.9										

* = Atmospheric %O2 Concentrations

Leak Test Log

Date	Intital Vacuum Reading	Time	Vacuum Reading After 10 Min.*	Time	Initials

*Min Minutes

Sundance Consulting Inc.

Purge Log

Project Name: Bulk Fuels Facility

Project Location: Kirtland Air Force Base

Well ID/Port Depth: _____

Samplers: _____

Sampler Signature/Date :_____

Screened Interval: Top:_____(ft. b.g.s)
Bottom:_____(ft. b.g.s)

Is Well damaged/Flooded: Yes - No

If yes, describe: _____

Weather Observations _____

Pump ID:_____ Horiba ID: _____ Sample System ID: _____ Manometer ID: _____

Was the sample system purged of hydrocarbons before connection with well port: Yes - No

Initial Static Pressure (inWC) _____

A. Pre- Calculated purge volume (cu. ft) Located on well data sheet	(Ft^3)
B. Flow Rate (SCFM) On flowmeter for air pump	(SCFM)
C. Purge time (Min. and Sec.) 1. Equals A/B 2. Write down whole number as minute 3. Multiple decimal by 60 for seconds	

Post Purge Static Pressure (inWC) _____

Sample Date/Time	CO2 %	O2%	Actual Purge Time	Hydrocarbons (ppmv)

Comments: _____

Bottle Vac. Number:_____

Initial B.V. Pressure:_____

Sample ID: _____

Final B.V. Pressure: _____

Reviewed by (Name): _____

Reviewer (Signature/Date): _____

Sundance Consulting Inc.

Date: _____

Time: _____

Project No.: __US01-023_____

C0C #: _____

Sample Collection Log

Project Name: ___KAFB_Bulk Fuels Facility_____

Sample No.: _____

Sample Location: ___Kirtland AFB ____

Sample Type: ____GRAB_____

Composite: (Y/N) _____No_____

Sample Team: _____

Trip Blank: _____

Sample:

Analytical Suite	Preservative	Container	TAT	Initials
VOCs by TO-15, EDB by CARB 422, Air-Phase Petroleum Hydrocarbons by MA APH 1.0, and Fixed Gases (H2, CO, CO2, N2, CH4 and O2/Ar) by E3C.	None	One Liter Amber Glass	15 Days	

Comments:

_____

_____

_____

_____

Logged By: _____ **Reviewed By**: _____

Air - Chain of Custody Record & Analytical Service Request

Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

COC #:

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

Company Name & Address (Reporting Information)

Sundance Consulting, Inc.
6700 Jefferson St NE, Suite C-3,
Albuquerque, NM 87109

Project Name/ Number: KAFB Bulk Fuels Facility/US01-023

Waynbill Number:

Project Manager: Rachel Hobbs

P.O. # / Billing Information:

Phone: 505-835-7660

Fax: 505-345-0742

Email Address for Result Reporting: rhobbs@sundance@sundance-inc.net

Sampler (Print & Sign):

Analytical Methods:

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Bottle Vac ID (Bar code # - AC, SC, etc.)	Flow Controller ID (Bar code # - FC #)	Bottle Vac Start Pressure "Hg	Bottle Vac End Pressure "Hg/psig	Sample Volume	TO-15 Project Specific List	MA APH HC Ranges	CARB 422 1,2-Dibromoethane	EPA 3C (O2, N2, CO2, CH4)	Comments e.g. Actual Preservative or specific instructions

Report Tier Levels - please select

Tier I - Results (Default if not specified) _____
Tier II (Results + QC Summaries) _____
Tier III (Results + QC & Calibration Summaries) _____
Tier IV (Data Validation Package) 10% Surcharge _____

EDD required Yes / No
Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:

Cooler / Blank Temperature ____°C

Sundance Consulting Inc.

Sample Collection Log

Date: _____

Time: _____

Project No: __US01-023_____

COC #:_____

Task:_____

Project Name ___KAFB Bulk Fuels Facility_____

Sample No. _____

Sample Location ___Kirtland AFB _____

Sample Type ____GRAB_____

Composite: Y/N ____No_____

Sample Team _____

Trip Blank _____

Sample:

Analytical Suite	Preservative	Quantity	Container	Temp.	FLT.	TAT	Initials
EDB by EPA 504.1	None	3	3x40 ml VOA	4°C	N	10 Days	
BTEX by EPA 524.2	HCL	3	3x40 ml VOA	4°C	N	10 Days	

Comments:

_____

_____

_____

_____

Logged by: _____ Reviewed by: _____

Groundwater- Chain of Custody Record & Analytical Service Request

Page _____ of _____

1317 S. 13th Ave
Kelso, WA 98626
Phone (360) 577 7222
Fax (360) 636 1068

Waybill #	COC #:

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

Company Name & Address (Reporting Information)	PO/Billing Information	Project Name / Project Number
Sundance Consulting, Inc. 6700 Jefferson St NE, Suite C-3, Albuquerque, NM 87109		KAFB Bulk Fuel Facilities US01-23
Project Manager: Rachel Hobbs	Sampler Name/Signature	Analytical Methods:
Phone: 505-835-7660 / Fax: 505-345-0742		
Email Address for Result Reporting: rhobbs@sundance@sundance-inc.net		

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Sample Matrix	No. Of Containers	Preservative					Sample Volume	Comments (e.g. specific instructions)

Report Tier Levels - please select

Tier I - Results (Default if not specified) _____
Tier II (Results + QC Summaries) _____
Tier III (Results + QC & Calibration Summaries) _____
Tier IV (Data Validation Package) 10% Surcharge _____

EDD required Yes / No
Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

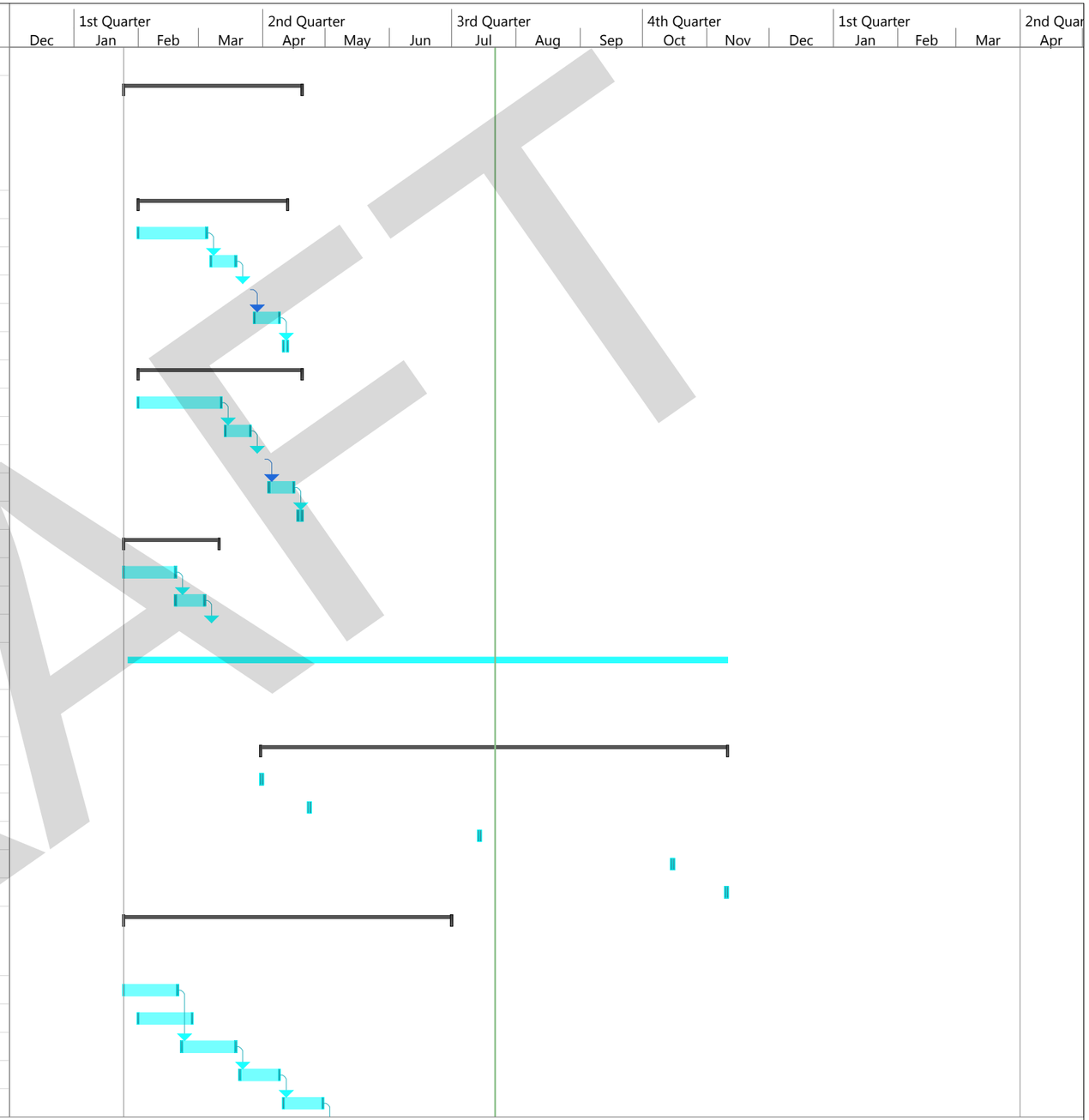
Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:		Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Date:	Time:		Received by: (Signature)	Date:	Time:

Cooler / Blank Temperature ____°C

Appendix B
Project Schedule

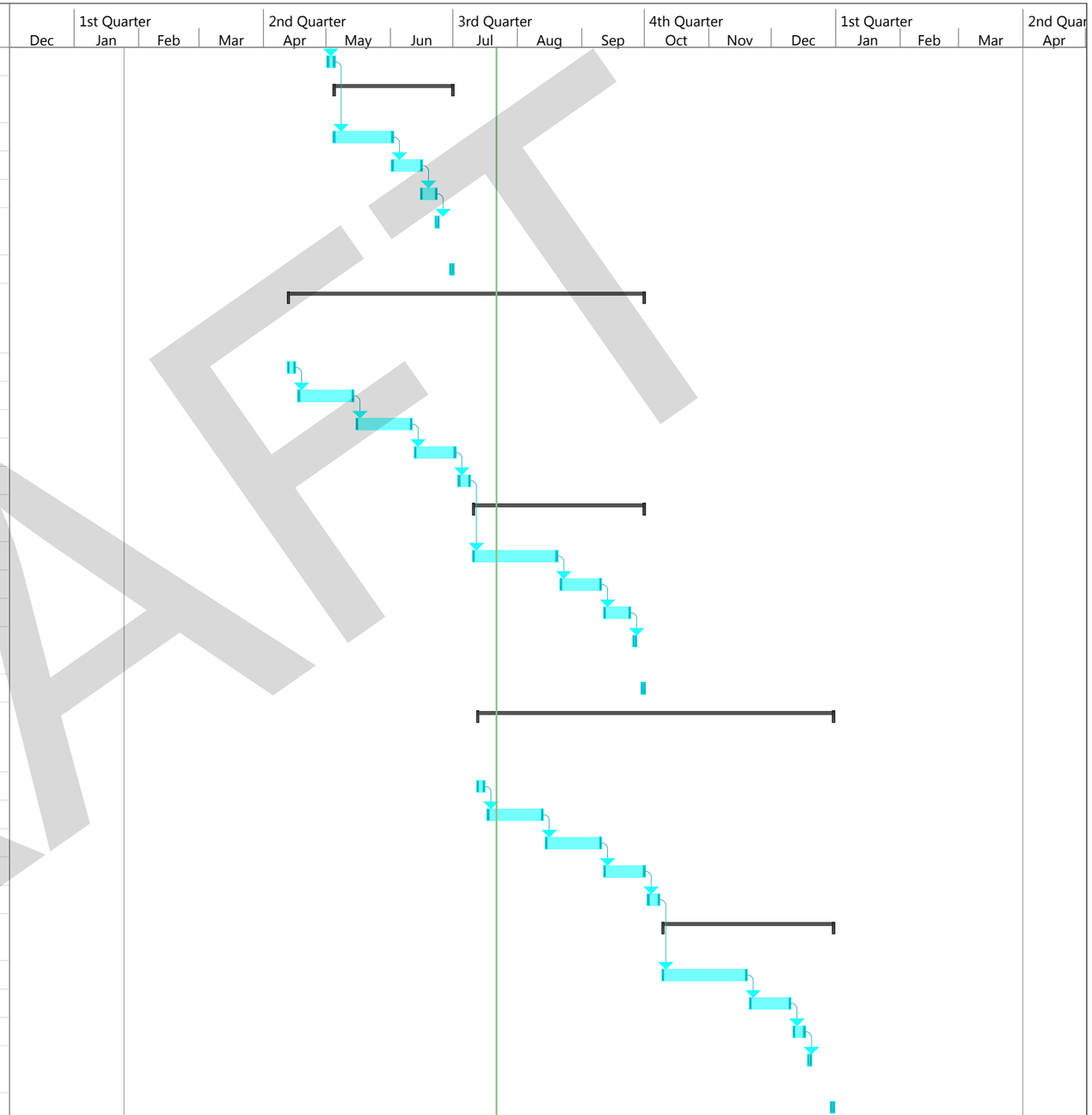
ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
1		Kick Off Meeting	1 day	Mon 2/1/16	Mon 2/1/16	
2		Update Quality Assurance Project Plan (Accident Prevention Plan (APP), Sampling and Analysis Plan (SAP), Field Sampling Plan (FSP)) CY 2016	62 days	Mon 1/25/16	Tue 4/19/16	
3		Work Plan	52 days	Mon 2/1/16	Tue 4/12/16	
4		Draft Work Plan	25 days	Mon 2/1/16	Fri 3/4/16	
5		USACE/AFCEC/KAFB Review	10 days	Mon 3/7/16	Fri 3/18/16	4
6		Incoporate Comments	5 days	Mon 3/21/16	Fri 3/25/16	5
7		Commander Signature	10 days	Mon 3/28/16	Fri 4/8/16	6
8		Delivery to NMED	2 days	Mon 4/11/16	Tue 4/12/16	7
9		QAPjP (Attached to Work Plan)	57 days	Mon 2/1/16	Tue 4/19/16	
10		Draft QAPjP	30 days	Mon 2/1/16	Fri 3/11/16	
11		USACE/AFCEC/KAFB Review	10 days	Mon 3/14/16	Fri 3/25/16	10
12		Incoporate Comments	5 days	Mon 3/28/16	Fri 4/1/16	11
13		Commander Signature	10 days	Mon 4/4/16	Fri 4/15/16	12
14		Delivery to NMED	2 days	Mon 4/18/16	Tue 4/19/16	13
15		APP	34 days	Mon 1/25/16	Thu 3/10/16	
16		Draft APP	19 days	Mon 1/25/16	Thu 2/18/16	
17		USACE/AFCEC/KAFB Review	10 days	Fri 2/19/16	Thu 3/3/16	16
18		Incoporate Comments	5 days	Fri 3/4/16	Thu 3/10/16	17
19		New Recurring Project Meetings CY 2016				
20		Status calls/CSI/other meetings to be held as needed				
21		Public Meetings CY 2016	161 days	Thu 3/31/16	Thu 11/10/16	
22		1st Public Meeting	1 day	Thu 3/31/16	Thu 3/31/16	
23		1st Field Trip	1 day	Sat 4/23/16	Sat 4/23/16	
24		2nd Public Meeting	1 day	Thu 7/14/16	Thu 7/14/16	
25		2nd Field Trip	1 day	Sat 10/15/16	Sat 10/15/16	
26		3rd Public Meeting	1 day	Thu 11/10/16	Thu 11/10/16	
27		Vadose Zone Sampling, Analysis and Reporting First Quarter CY 2016	114 days	Mon 1/25/16	Thu 6/30/16	
28		Project Mobilization	20 days	Mon 1/25/16	Fri 2/19/16	
29		Sample Planning	20 days	Mon 2/1/16	Fri 2/26/16	
30		Soil Vapor Sampling Field Work	20 days	Mon 2/22/16	Fri 3/18/16	28
31		Laboratory Analysis	15 days	Mon 3/21/16	Fri 4/8/16	30
32		Data Validation	15 days	Mon 4/11/16	Fri 4/29/16	31



Project: KAFB_Schedule_072216
Date: Fri 7/22/16

Task	Project Summary	Manual Task	Start-only	Deadline	
Split	Inactive Task	Duration-only	Finish-only	Progress	
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
33		Database Management	3 days	Mon 5/2/16	Wed 5/4/16	32
34		Data Dump Report First Quarter CY 2016	41 days	Thu 5/5/16	Thu 6/30/16	
35		Draft Data Dump Report	20 days	Thu 5/5/16	Wed 6/1/16	33
36		USACE/AFCEC/KAFB Review	10 days	Thu 6/2/16	Wed 6/15/16	35
37		Incoporporate Comments	5 days	Thu 6/16/16	Wed 6/22/16	36
38		Incorporation into Quarterly Report	1 day	Thu 6/23/16	Thu 6/23/16	37
39		Delivery to NMED	1 day	Thu 6/30/16	Thu 6/30/16	
40		Vadose Zone Sampling, Analysis and Reporting Second Quarter CY 2016	123 days	Wed 4/13/16	Fri 9/30/16	
41		Sample Planning	3 days	Wed 4/13/16	Fri 4/15/16	
42		Soil Vapor Sampling Field Work	20 days	Mon 4/18/16	Fri 5/13/16	41
43		Laboratory Analysis	20 days	Mon 5/16/16	Fri 6/10/16	42
44		Data Validation	15 days	Mon 6/13/16	Fri 7/1/16	43
45		Database Management	5 days	Mon 7/4/16	Fri 7/8/16	44
46		Data Dump Report Second Quarter CY 2016	60 days	Mon 7/11/16	Fri 9/30/16	
47		Draft Data Dump Report	30 days	Mon 7/11/16	Fri 8/19/16	45
48		USACE/AFCEC/KAFB Review	15 days	Mon 8/22/16	Fri 9/9/16	47
49		Incoporporate Comments	10 days	Mon 9/12/16	Fri 9/23/16	48
50		Incorporation into Quarterly Report	1 day	Mon 9/26/16	Mon 9/26/16	49
51		Delivery to NMED	1 day	Fri 9/30/16	Fri 9/30/16	
52		Vadose Zone Sampling, Analysis and Reporting Third Quarter CY 2016	123 days	Wed 7/13/16	Fri 12/30/16	
53		Sample Planning	3 days	Wed 7/13/16	Fri 7/15/16	
54		Soil Vapor Sampling Field Work	20 days	Mon 7/18/16	Fri 8/12/16	53
55		Laboratory Analysis	20 days	Mon 8/15/16	Fri 9/9/16	54
56		Data Validation	15 days	Mon 9/12/16	Fri 9/30/16	55
57		Database Management	5 days	Mon 10/3/16	Fri 10/7/16	56
58		Data Dump Report Third Quarter CY 2016	60 days	Mon 10/10/16	Fri 12/30/16	
59		Draft Data Dump Report	30 days	Mon 10/10/16	Fri 11/18/16	57
60		USACE/AFCEC/KAFB Review	15 days	Mon 11/21/16	Fri 12/9/16	59
61		Incoporporate Comments	5 days	Mon 12/12/16	Fri 12/16/16	60
62		Incorporation into Quarterly Report	1 day	Mon 12/19/16	Mon 12/19/16	61
63		Delivery to NMED	1 day	Fri 12/30/16	Fri 12/30/16	



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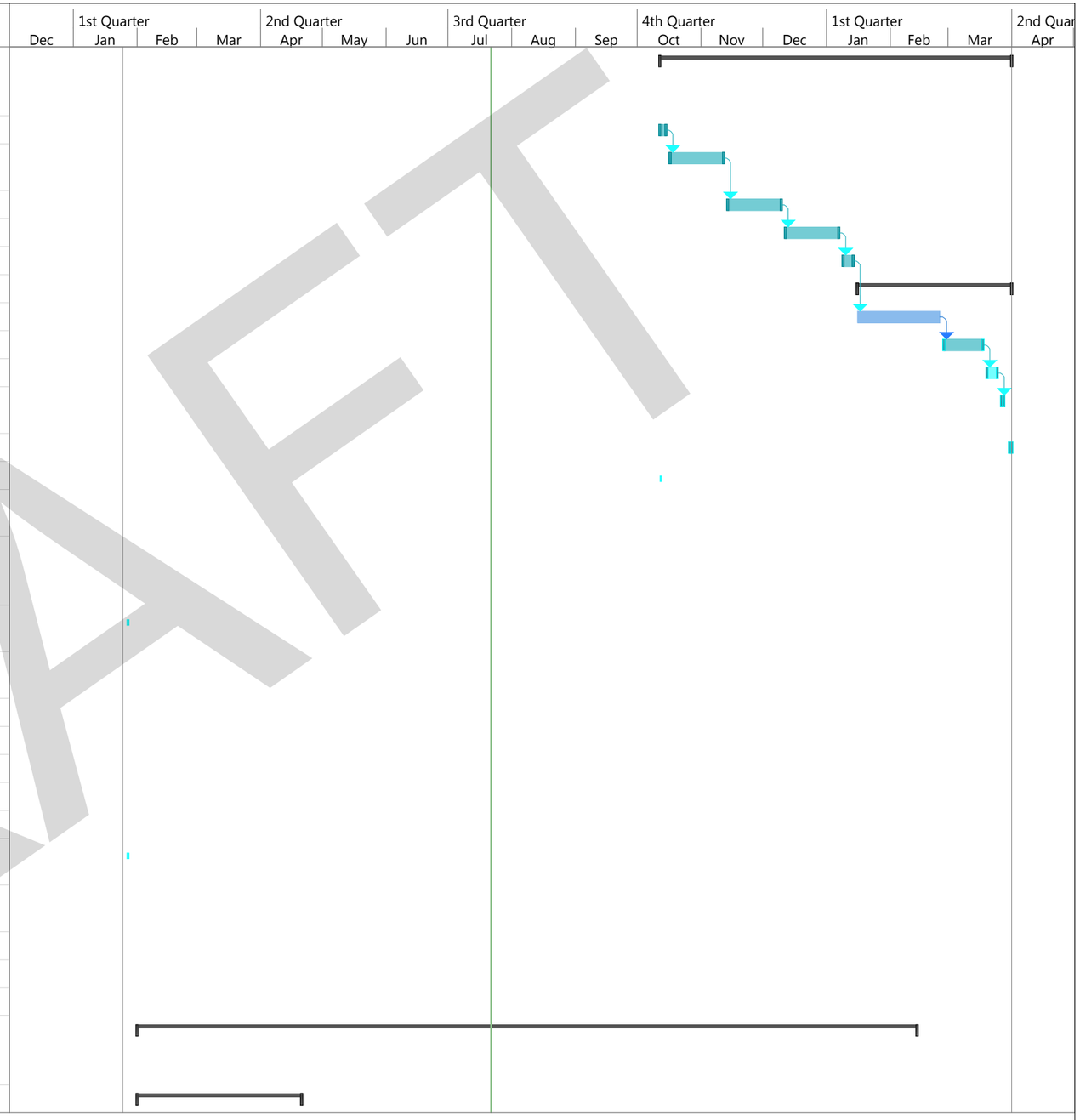
Date: Fri 7/22/16

Task	Project Summary	Manual Task	Start-only	Deadline	
Split	Inactive Task	Duration-only	Finish-only	Progress	
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
64		Vadose Zone Sampling, Analysis and Reporting Fourth Quarter CY 2016	123 days	Wed 10/12/16	Fri 3/31/17	
65		Sample Planning	3 days	Wed 10/12/16	Fri 10/14/16	
66		Soil Vapor Sampling Field Work	20 days	Mon 10/17/16	Fri 11/11/16	65
67		Laboratory Analysis	20 days	Mon 11/14/16	Fri 12/9/16	66
68		Data Validation	20 days	Mon 12/12/16	Fri 1/6/17	67
69		Database Management	5 days	Mon 1/9/17	Fri 1/13/17	68
70		Annual Monitoring Report	55 days	Mon 1/16/17	Fri 3/31/17	
71		Draft Annual Monitoring Report	30 days	Mon 1/16/17	Fri 2/24/17	69
72		USACE/AFCEC/KAFB Review	15 days	Mon 2/27/17	Fri 3/17/17	71
73		Incoporporate Comments	5 days	Mon 3/20/17	Fri 3/24/17	72
74		Incorporation into Quarterly Report	1 day	Mon 3/27/17	Mon 3/27/17	73
75		Delivery to NMED	1 day	Fri 3/31/17	Fri 3/31/17	
76		Maintenance Activities				
77		To be scheduled as determined necessary				
78		Well Installation and Abandonment - To be scheduled as determined necessary				
79		Well Installation				
80		Office of State Engineer Permit Application				
81		Kirtland Dig Permit				
82		Well Installation				
83		IDW Sampling and Letters				
84		OSE Report-Installation				
85		Well Survey				
86		Well Abandonment				
87		Office of State Engineer Permit Application				
88		Well Abandonment				
89		OSE Report-Abandonment				
90		Reporting				
91		Monthly Drinking Water Sampling, Analysis and Reporting CY 2016	271 days	Mon 2/1/16	Mon 2/13/17	
92		Month 1 (Feb 2016)	58 days	Mon 2/1/16	Wed 4/20/16	



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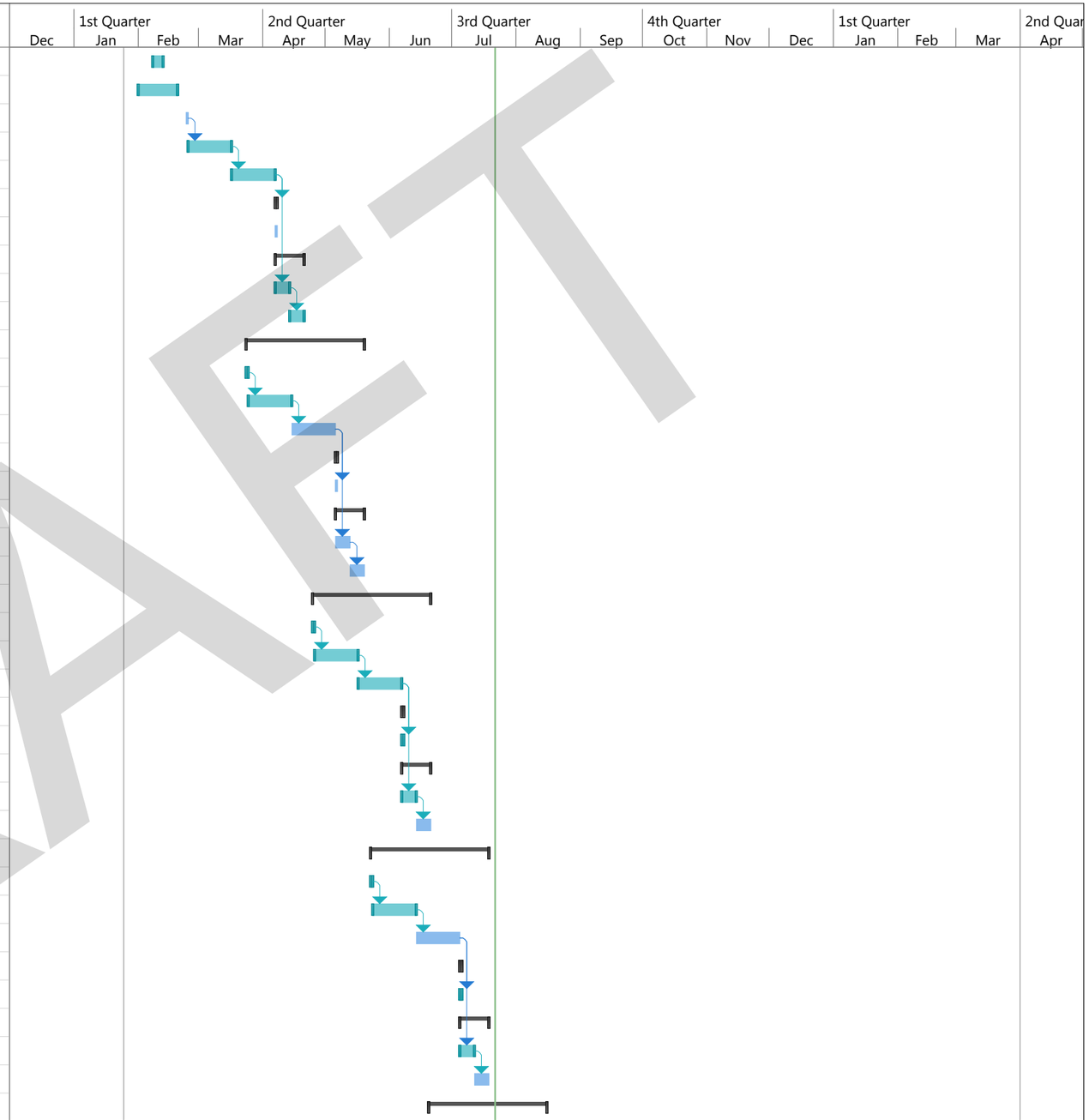
Task	Project Summary	Manual Task	Start-only	Deadline	
Split	Inactive Task	Duration-only	Finish-only	Progress	
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
93		Mobilization	5 days	Mon 2/8/16	Fri 2/12/16	
94		Sample Planning	15 days	Mon 2/1/16	Fri 2/19/16	
95		Field Work	1 day	Wed 2/24/16	Wed 2/24/16	
96		Laboratory Analysis	15 days	Thu 2/25/16	Wed 3/16/16	95
97		Data Validation	15 days	Thu 3/17/16	Wed 4/6/16	96
98		Database Management	1 day	Thu 4/7/16	Thu 4/7/16	97
99		ERPIMS uploads	1 day	Thu 4/7/16	Thu 4/7/16	
100		Technical Memo	10 days	Thu 4/7/16	Wed 4/20/16	
101		Draft Technical Memo	5 days	Thu 4/7/16	Wed 4/13/16	97
102		Submit To USACE	5 days	Thu 4/14/16	Wed 4/20/16	101
103		Month 2 (March 2016)	41 days	Thu 3/24/16	Thu 5/19/16	
104		Field Work	1 day	Thu 3/24/16	Thu 3/24/16	
105		Laboratory Analysis	15 days	Fri 3/25/16	Thu 4/14/16	104
106		Data Validation	15 days	Fri 4/15/16	Thu 5/5/16	105
107		Database Management	1 day	Fri 5/6/16	Fri 5/6/16	
108		ERPIMS uploads	1 day	Fri 5/6/16	Fri 5/6/16	106
109		Technical Memo	10 days	Fri 5/6/16	Thu 5/19/16	
110		Draft Technical Memo	5 days	Fri 5/6/16	Thu 5/12/16	106
111		USACE/AFCEC/KAFB Review	5 days	Fri 5/13/16	Thu 5/19/16	110
112		Month 3 (April 2016)	41 days	Mon 4/25/16	Mon 6/20/16	
113		Field Work	1 day	Mon 4/25/16	Mon 4/25/16	
114		Laboratory Analysis	15 days	Tue 4/26/16	Mon 5/16/16	113
115		Data Validation	15 days	Tue 5/17/16	Mon 6/6/16	114
116		Database Management	1 day	Tue 6/7/16	Tue 6/7/16	
117		ERPIMS uploads	1 day	Tue 6/7/16	Tue 6/7/16	115
118		Technical Memo	10 days	Tue 6/7/16	Mon 6/20/16	
119		Draft Technical Memo	5 days	Tue 6/7/16	Mon 6/13/16	115
120		USACE/AFCEC/KAFB Review	5 days	Tue 6/14/16	Mon 6/20/16	119
121		Month 4 (May 2016)	41 days	Mon 5/23/16	Mon 7/18/16	
122		Field Work	1 day	Mon 5/23/16	Mon 5/23/16	
123		Laboratory Analysis	15 days	Tue 5/24/16	Mon 6/13/16	122
124		Data Validation	15 days	Tue 6/14/16	Mon 7/4/16	123
125		Database Management	1 day	Tue 7/5/16	Tue 7/5/16	
126		ERPIMS uploads	1 day	Tue 7/5/16	Tue 7/5/16	124
127		Technical Memo	10 days	Tue 7/5/16	Mon 7/18/16	
128		Draft Technical Memo	5 days	Tue 7/5/16	Mon 7/11/16	124
129		USACE/AFCEC/KAFB Review	5 days	Tue 7/12/16	Mon 7/18/16	128
130		Month 5 (June 2016)	41 days	Mon 6/20/16	Mon 8/15/16	



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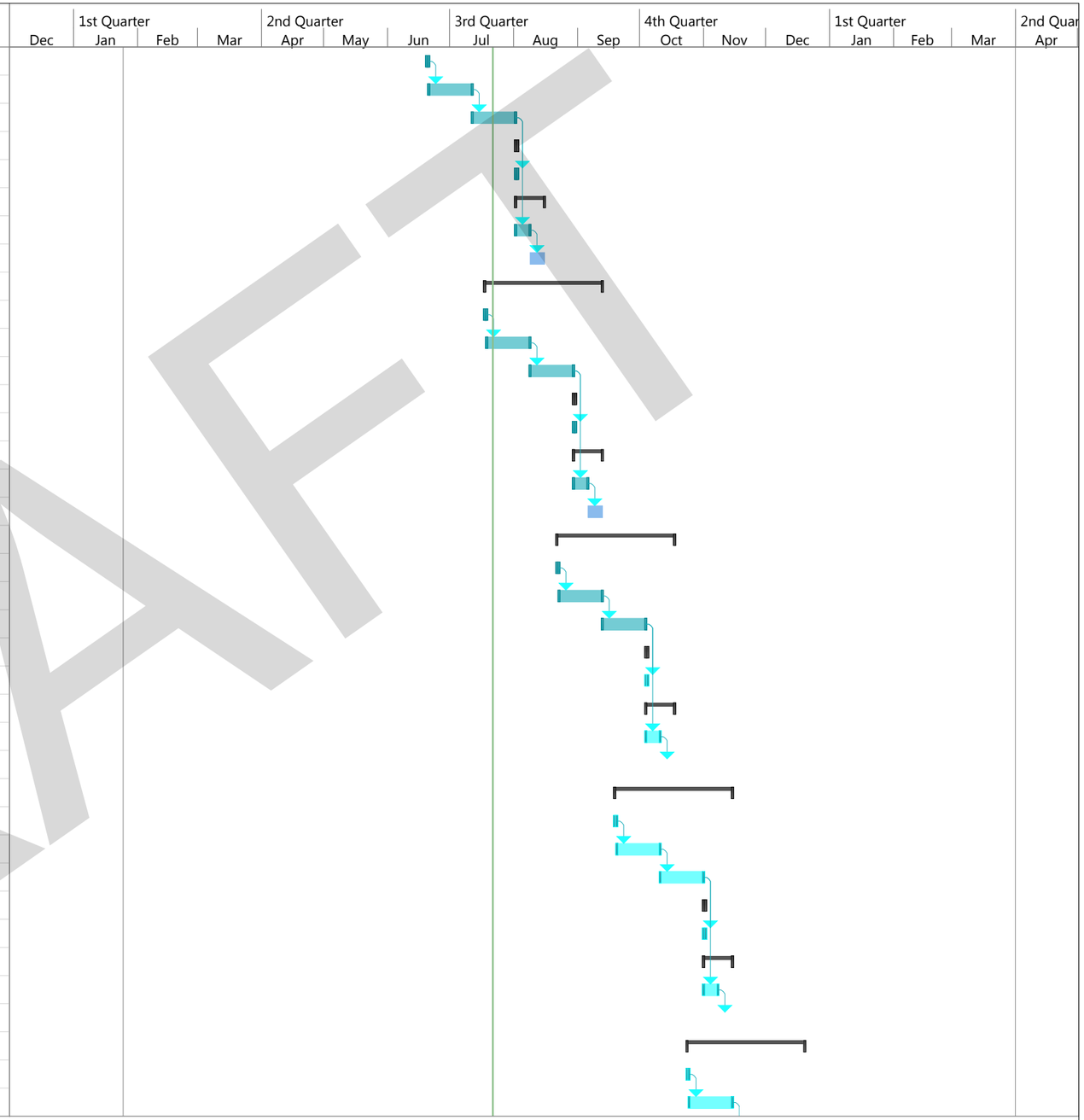
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
131		Field Work	1 day	Mon 6/20/16	Mon 6/20/16	
132		Laboratory Analysis	15 days	Tue 6/21/16	Mon 7/11/16	131
133		Data Validation	15 days	Tue 7/12/16	Mon 8/1/16	132
134		Database Management	1 day	Tue 8/2/16	Tue 8/2/16	
135		ERPIMS uploads	1 day	Tue 8/2/16	Tue 8/2/16	133
136		Technical Memo	10 days	Tue 8/2/16	Mon 8/15/16	
137		Draft Technical Memo	5 days	Tue 8/2/16	Mon 8/8/16	133
138		USACE/AFCEC/KAFB Review	5 days	Tue 8/9/16	Mon 8/15/16	137
139		Month 6 (July 2016)	41 days	Mon 7/18/16	Mon 9/12/16	
140		Field Work	1 day	Mon 7/18/16	Mon 7/18/16	
141		Laboratory Analysis	15 days	Tue 7/19/16	Mon 8/8/16	140
142		Data Validation	15 days	Tue 8/9/16	Mon 8/29/16	141
143		Database Management	1 day	Tue 8/30/16	Tue 8/30/16	
144		ERPIMS uploads	1 day	Tue 8/30/16	Tue 8/30/16	142
145		Technical Memo	10 days	Tue 8/30/16	Mon 9/12/16	
146		Draft Technical Memo	5 days	Tue 8/30/16	Mon 9/5/16	142
147		USACE/AFCEC/KAFB Review	5 days	Tue 9/6/16	Mon 9/12/16	146
148		Month 7 (August 2016)	41 days	Mon 8/22/16	Mon 10/17/16	
149		Field Work	1 day	Mon 8/22/16	Mon 8/22/16	
150		Laboratory Analysis	15 days	Tue 8/23/16	Mon 9/12/16	149
151		Data Validation	15 days	Tue 9/13/16	Mon 10/3/16	150
152		Database Management	1 day	Tue 10/4/16	Tue 10/4/16	
153		ERPIMS uploads	1 day	Tue 10/4/16	Tue 10/4/16	151
154		Technical Memo	10 days	Tue 10/4/16	Mon 10/17/16	
155		Draft Technical Memo	5 days	Tue 10/4/16	Mon 10/10/16	151
156		USACE/AFCEC/KAFB Review	5 days	Tue 10/11/16	Mon 10/17/16	155
157		Month 8 (September 2016)	41 days	Mon 9/19/16	Mon 11/14/16	
158		Field Work	1 day	Mon 9/19/16	Mon 9/19/16	
159		Laboratory Analysis	15 days	Tue 9/20/16	Mon 10/10/16	158
160		Data Validation	15 days	Tue 10/11/16	Mon 10/31/16	159
161		Database Management	1 day	Tue 11/1/16	Tue 11/1/16	
162		ERPIMS uploads	1 day	Tue 11/1/16	Tue 11/1/16	160
163		Technical Memo	10 days	Tue 11/1/16	Mon 11/14/16	
164		Draft Technical Memo	5 days	Tue 11/1/16	Mon 11/7/16	160
165		USACE/AFCEC/KAFB Review	5 days	Tue 11/8/16	Mon 11/14/16	164
166		Month 9 (October 2016)	41 days	Mon 10/24/16	Mon 12/19/16	
167		Field Work	1 day	Mon 10/24/16	Mon 10/24/16	
168		Laboratory Analysis	15 days	Tue 10/25/16	Mon 11/14/16	167



Project: KAFB_Schedule_072216
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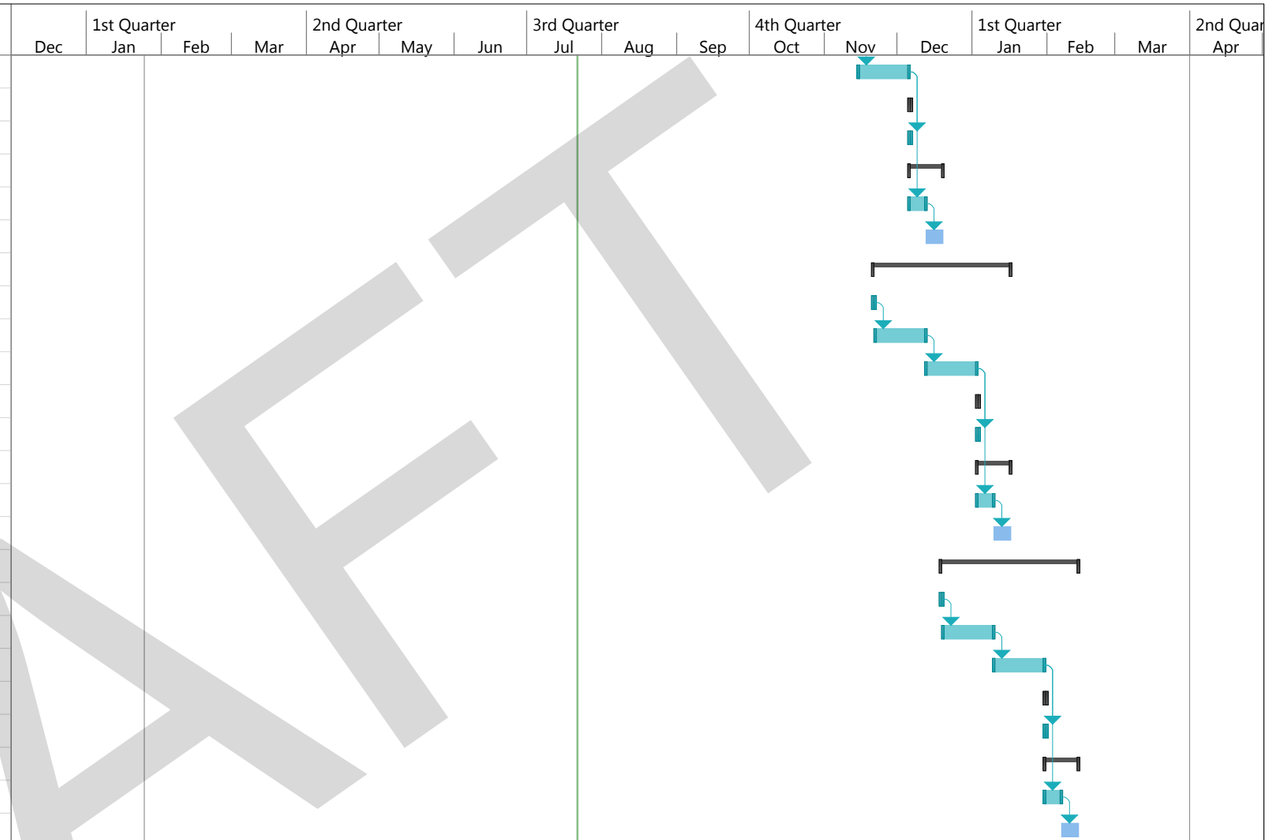
Task	Project Summary	Manual Task	Start-only	Deadline	
Split	Inactive Task	Duration-only	Finish-only	Progress	
Milestone	Inactive Milestone	Manual Summary Rollup	External Tasks	Manual Progress	
Summary	Inactive Summary	Manual Summary	External Milestone		

ID	Task Mode	Task Name	Duration	Start	Finish	Predecessors
169		Data Validation	15 days	Tue 11/15/16	Mon 12/5/16	168
170		Database Management	1 day	Tue 12/6/16	Tue 12/6/16	
171		ERPIMS uploads	1 day	Tue 12/6/16	Tue 12/6/16	169
172		Technical Memo	10 days	Tue 12/6/16	Mon 12/19/16	
173		Draft Technical Memo	5 days	Tue 12/6/16	Mon 12/12/16	169
174		USACE/AFCEC/KAFB Review	5 days	Tue 12/13/16	Mon 12/19/16	173
175		Month 10 (November 2016)	41 days	Mon 11/21/16	Mon 1/16/17	
176		Field Work	1 day	Mon 11/21/16	Mon 11/21/16	
177		Laboratory Analysis	15 days	Tue 11/22/16	Mon 12/12/16	176
178		Data Validation	15 days	Tue 12/13/16	Mon 1/2/17	177
179		Database Management	1 day	Tue 1/3/17	Tue 1/3/17	
180		ERPIMS uploads	1 day	Tue 1/3/17	Tue 1/3/17	178
181		Technical Memo	10 days	Tue 1/3/17	Mon 1/16/17	
182		Draft Technical Memo	5 days	Tue 1/3/17	Mon 1/9/17	178
183		USACE/AFCEC/KAFB Review	5 days	Tue 1/10/17	Mon 1/16/17	182
184		Month 11 (December 2016)	41 days	Mon 12/19/16	Mon 2/13/17	
185		Field Work	1 day	Mon 12/19/16	Mon 12/19/16	
186		Laboratory Analysis	15 days	Tue 12/20/16	Mon 1/9/17	185
187		Data Validation	15 days	Tue 1/10/17	Mon 1/30/17	186
188		Database Management	1 day	Tue 1/31/17	Tue 1/31/17	
189		ERPIMS uploads	1 day	Tue 1/31/17	Tue 1/31/17	187
190		Technical Memo	10 days	Tue 1/31/17	Mon 2/13/17	
191		Draft Technical Memo	5 days	Tue 1/31/17	Mon 2/6/17	187
192		USACE/AFCEC/KAFB Review	5 days	Tue 2/7/17	Mon 2/13/17	191
193		Monthly Drinking Water Sampling, Analysis and Reporting CY 2017				
194		Will follow same schedule as 2016 events				
195		Vadose Zone Sampling, Analysis and Reporting First Quarter CY 2017				
196		Will follow similar schedule to 2016 events				



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Summary	Inactive Summary	Manual Summary	External Milestone	

Appendix C
Quality Assurance Project Plan

Quality Assurance Project Plan to be delivered separately.

KIRTLAND AIR FORCE BASE
ALBUQUERQUE, NEW MEXICO

Quality Assurance Project Plan for Soil Vapor Monitoring and Drinking Water Monitoring Bulk Fuels Facility Solid Waste Management Unit ST-106/SS-111 Kirtland Air Force Base, New Mexico

August ~~st April~~ 2016

Prepared for

U.S. Army Corps of Engineers
Albuquerque District
4101 Jefferson Plaza NE
Albuquerque, NM 87109

Contract No. W912PP-16-C-0002

Prepared by

Sundance Consulting, Inc.
8210 Louisiana Blvd. NE Suite C
Albuquerque, NM 87113

DISTRIBUTION LIST
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

QAPjP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address
Amy Sanchez	USACE Albuquerque District COR	USACE	505-342-3234	Amy.E.Sanchez@usace.army.mil
Trent Simpler, P.E.	USACE Albuquerque District PM	USACE	505-342-4823	Trent.Simpler@usace.army.mil
Mark Phaneuf, P.G.	USACE Albuquerque District Technical POC	USACE	505-342-3295	Mark.J.Phaneuf@usace.army.mil
Adria Bodour, PhD	AFCEC Technical Lead	AFCEC	210-748-4035	adria.bodour.1@us.af.mil
Wayne Bitner	KAFB Environmental Restoration Lead	AFCEC	505-853-3484	Ludie.Bitner@us.af.mil
NMED	Regulator	NMED Hazardous Waste Bureau	505-428-2500	2905 Rodeo Park Drive E#1, Santa Fe, New Mexico 87505
James Lockhart, P.E.	Sundance Vice President of Operations	Sundance	208-233-2929	jlockhart@sundance-inc.net
~~Patrick Scher~~ Rachel Hobbs, P.G.	Sundance PM/SSHO	Sundance	505-835-7660 ~~x154~~x159	~~pscher@sundance-inc.net~~ rhobbs@sundance-inc.net
~~Rachel Hobbs~~ Eric Klingel, PhD, P.G.	Sundance Project ~~Geologist/SSHO~~ Technical Lead	Sundance	505-835-7660 ~~x159~~x156	~~rhobbs@sundance-inc.net~~ eklingel@sundance-inc.net
Ryan Wortman	Sundance Field Team Lead/Deputy SSHO	Sundance	505-835-7660 x155	rwortman@sundance-inc.net

DISTRIBUTION LIST
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded)

Acronyms and Abbreviations:
AFCEC = Air Force Civil Engineer Center
BFF = Bulk Fuels Facility
COR = Contracting Officer's Representative
KAFB = Kirtland Air Force Base
NMED = New Mexico Environment Department
P.E. = Professional Engineer
P.G. = Professional Geologist
PhD = Doctor of Philosophy
PM = Project Manager
POC = point of contact
QAPjP = Quality Assurance Project Plan
SSHO = Site Safety and Health Officer
Sundance = Sundance Consulting, Inc.
USACE = U.S. Army Corps of Engineers

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ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
%	percent
AFB	Air Force Base
ALS	ALS Environmental Laboratory
APH	air-phase petroleum hydrocarbon
APP	Accident Prevention Plan
ASTM	ASTM International
BFF	Bulk Fuels Facility
bgs	below ground surface
BTEX	benzene, toluene, ethylbenzene, and xylenes
CARB	California Air Resources Board
CO	carbon monoxide
COA	City of Albuquerque
CoC	contaminants of concern
COC	chain-of-custody
COPC	contaminant of potential concern
CY	calendar year
DoD	U.S. Department of Defense
DQA	data quality assessment
DQO	data quality objective
EDB	ethylene dibromide
e.g.	example given
ELAP	Environmental Laboratory Accreditation Program
EPA	U.S. Environmental Protection Agency
ft	foot/feet
HC	hydrocarbon
ID	identification
IDW	investigation-derived waste
KAFB	Kirtland Air Force Base
L	liter
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDC	Laboratory Data Consultants, Inc.
LOQ	limit of quantitation
MA APH	Massachusetts Air-Phase Petroleum Hydrocarbons
MA DEP	Massachusetts Department of Environmental Protection

ACRONYMS AND ABBREVIATIONS (CONTINUED)

MCL	maximum contaminant level
MDL	method detection limit
mL	milliliter
MS	matrix spike
MSD	matrix spike duplicate
NAPL	non-aqueous phase liquid
NCR	Nonconformance Report
NIST	National Institute of Standards and Technology
NMED	New Mexico Environment Department
NMWQCC	New Mexico Water Quality Control Commission
No.	number
NOD	Notice of Deficiency
O_2	oxygen (molecular)
OSRTI	Office of Superfund Remediation and Technology Innovation
OSWER	Office of Solid Waste and Emergency Response
PARCC	precision, accuracy, representiveness, comparability and completeness
P.E.	Professional Engineer
P.G.	Professional Geologist
PM	Project Manager
POC	point of contact
PPE	personal protective equipment
QA	quality assurance
QAPjP	Quality Assurance Project Plan
QC	quality control
QCM	Quality Control Manager
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
RPD	relative percent difference
RSL	regional screening level
Sundance	Sundance Consulting, Inc.
SOP	standard operating procedure
SVE	soil vapor extraction
SVM	soil vapor monitoring
SVMP	soil vapor monitoring point
SVOC	semi volatile organic compound
SWMU	solid waste management unit
TPH	total petroleum hydrocarbons
U.S.	United States
USACE	U.S. Army Corps of Engineers
USAF	U.S. Air Force

ACRONYMS AND ABBREVIATIONS (CONCLUDED)

VA	Veteran's Administration
VOC	volatile organic compound
VPH	volatile petroleum hydrocarbon
WP	Work Plan

SIGNATURE SHEET

SOIL VAPOR MONITORING AND DRINKING WATER MONITORING BULK FUELS FACILITY, SOLID WASTE MANAGEMENT UNIT ST-106/SS-111 KIRTLAND AIR FORCE BASE, NEW MEXICO

~~April 22, 2016~~July 22, 2016

Ryan Wortman,~~Rachel Hobbs, P.G.~~
Date

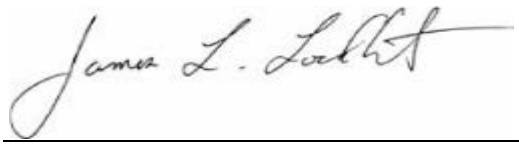
Field Team Lead/ Deputy Site Safety and Health Officer~~/Project Geologist~~
Sundance Consulting, Inc.

~~April~~ July 22, 2016

Rachel Hobbs~~Patrick Scher~~, P.G.
Date

Project Manager
Sundance Consulting, Inc.

CERTIFICATION



~~April~~ July 22, 2016

James L. Lockhart, P.E.
Date

Vice President of Operations
Sundance Consulting Inc.

EXECUTIVE SUMMARY

This Quality Assurance Project Plan (QAPjP) has been prepared by Sundance Consulting, Inc. (Sundance) under the U.S. Army Corps of Engineers (USACE)–Albuquerque District, Contract Number W912PP-16-C-0002. This QAPjP was developed to support soil vapor monitoring and drinking water monitoring at Kirtland Air Force Base (KAFB). The work to be conducted under this contract will include periodic monitoring of soil vapor and drinking water, as well as the abandonment and installation of soil vapor monitoring points at a location and date to be determined by USACE.

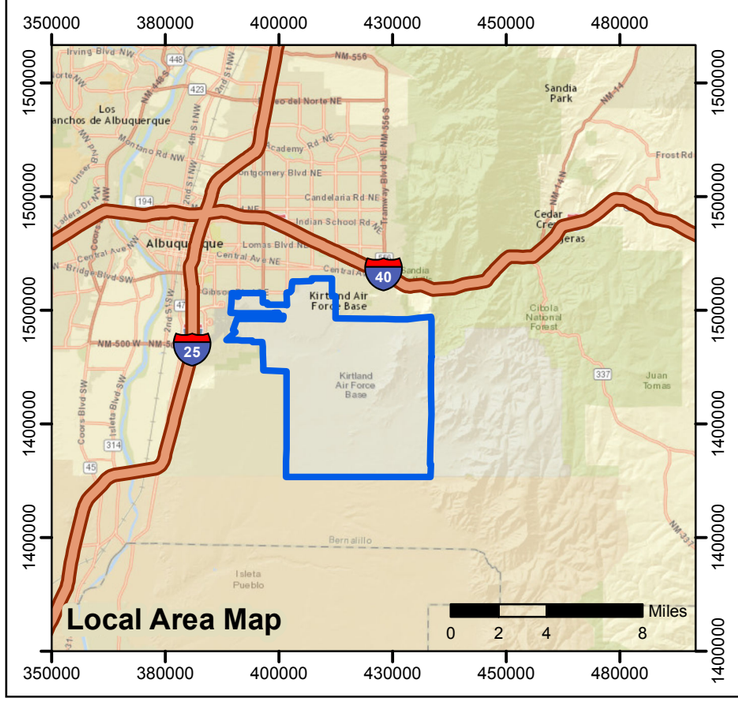
This QAPjP was developed for the periodic monitoring at KAFB Bulk Fuels Facility to meet the quality control requirements defined in the Department of Defense Quality Systems Manual (Version 5.0, July 2013). The QAPjP documents project management procedures and describes data generation and acquisition for field sampling and laboratory analytical processes, laboratory analytical methods, quality assurance/quality control protocols, data validation and usability, assessment and oversight, data management processes, and reporting requirements to be implemented for the project.

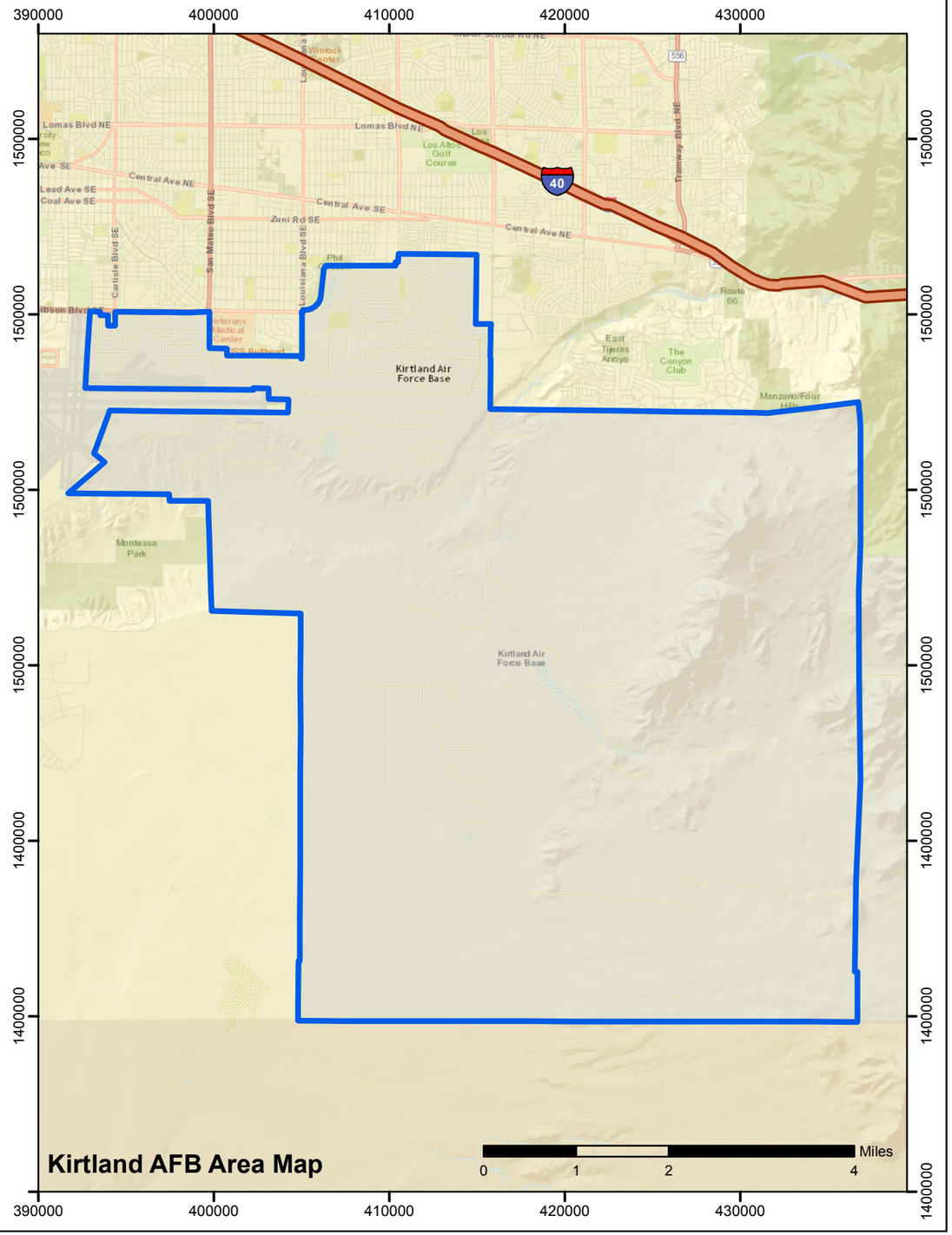
1 INTRODUCTION

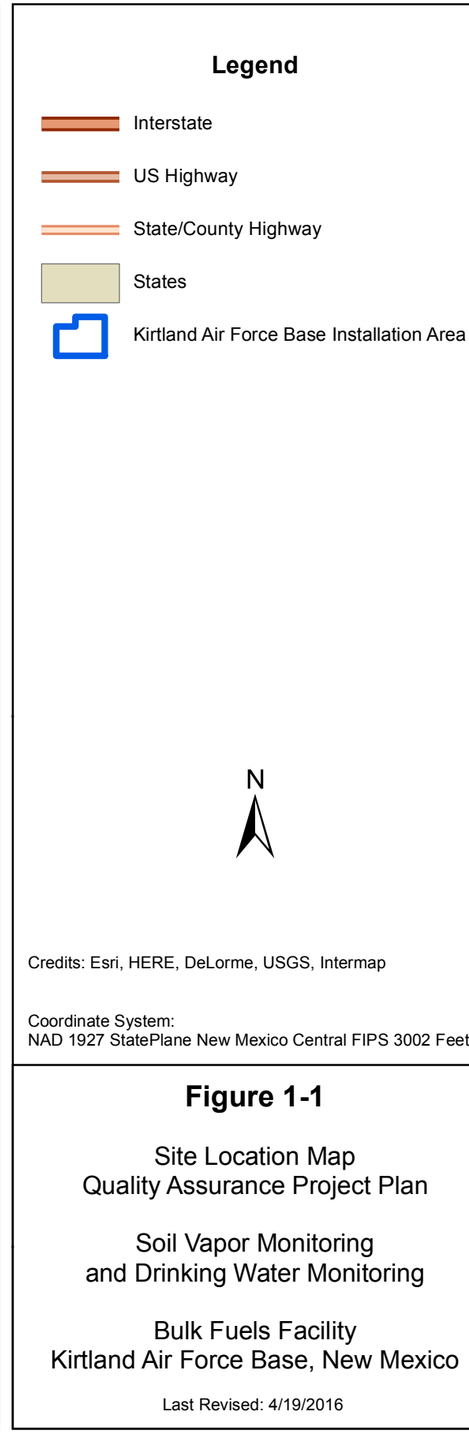
This Soil Vapor Monitoring and Drinking Water Monitoring Quality Assure Project Plan was prepared by Sundance Consulting, Inc. (Sundance) for the United States Army Corps of Engineers (USACE) under contract number W912PP-16-C-0002. Kirtland Air Force Base (KAFB) is located in Bernalillo County, in central New Mexico, southeast of and adjacent to the City of Albuquerque and the Albuquerque International Sunport. The approximate area of the base is 52,287 acres. The Bulk Fuels Facility (BFF) site, comprised of Solid Waste Management Unit (SWMU) ST-106/SS-111, is located in the northwestern part of KAFB (Figure 1-1). Environmental restoration efforts at the BFF site are being conducted under requirements set forth in the Resource Conservation and Recovery Act (RCRA), Permit Number (No.) NM9570024423, with the New Mexico Environment Department (NMED) serving as the lead regulatory agency (NMED 2010). This Quality Assurance Project Plan (QAPjP) addresses activities that are continuing the implementation of the RCRA Interim Measures for the site, including continuation of soil vapor monitoring (SVM) and drinking water supply well monitoring, and the installation and abandonment of one or more soil vapor monitoring points (SVMPs) at locations and dates to be determined by USACE with United States Air Force (USAF) input.

The BFF and associated infrastructure operated from 1953 through 1999. During this time, the fueling area was separated into a tank holding area where bulk shipments of fuel were received, and a fuel loading area where individual fuel railcars or trucks were emptied or disharged. In 1999, KAFB stopped using the underground piping at the facility and removed this piping from service due to discovery of a leak. Although the fuel leak was identified by KAFB, the exact history of the releases is unknown. Releases could have occurred when fuel was transferred from railcars or trucks to the pump house. Initially, it was thought that the leak only affected surface soil around the identified source area; however, KAFB learned through characterization activities that the leaked fuel had migrated to the groundwater table and that dissolved phase fuel contamination had migrated northeast and north of KAFB.

In order to comply with NMED Hazardous Waste Bureau requirements, a RCRA Facility Investigation (RFI) has been ongoing since 2011. As part of this ongoing investigation, 284 SVMPs have been and are sampled to characterize the nature and extent of soil vapor contamination in the vadose zone (approximately 460 feet (ft) from the ground surface to the top of the water table). In addition, drinking water supply wells have been and continue to be monitored to ensure they have not been impacted by contaminants from the BFF site. Under this project, SVM will continue and annual maintenance will be performed at the SVMPs. The drinking water supply well monitoring will continue through sampling and analysis.







Credits: Esri, HERE, DeLorme, USGS, Intermap

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

Figure 1-1

Site Location Map
Quality Assurance Project Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016

2 PROJECT MANAGEMENT AND ORGANIZATION

Project management for the soil vapor and drinking water monitoring activities will be performed in accordance with the requirements and the authority of the USACE, Contract No. W912PP-16-C-0002, and other applicable federal and state regulations.

The BFF project team consists of representatives from USACE, USAF, Sundance and its subcontractors, and the NMED. The USAF is the lead federal agency for direction of site activities and decision-making. The NMED Hazardous Waste Bureau is the lead regulatory agency.

2.1 Project Quality Assurance Organization

The project quality assurance (QA) organization, presented in Figure 2-1, identifies key Sundance individuals and responsibilities to ensure project QA objectives are achieved for soil vapor and drinking water monitoring.

2.2 Personnel Qualifications

Personnel qualifications for key Sundance individuals supporting the monitoring activities are listed in Table 2-1 in addition to the title, responsibility, education, experience, and authority level.

2.3 Task Description

The tasks to be addressed under this QAPjP include quarterly SVM and monthly drinking water supply well monitoring. This document addresses all of the quality aspects of the following tasks:

- Sample SVM network of 284 SVMPs quarterly for volatile organic compounds (VOCs) by method TO-15, ethylene dibromide (EDB) by method California Air Resources Board (CARB) 422, air-phase petroleum hydrocarbons (APH) by Massachusetts Air-Phase Petroleum Hydrocarbon (MA APH) method 1.0, and fixed gases by U.S. Environmental Protection Agency (EPA) method 3C (Figure 2-2).

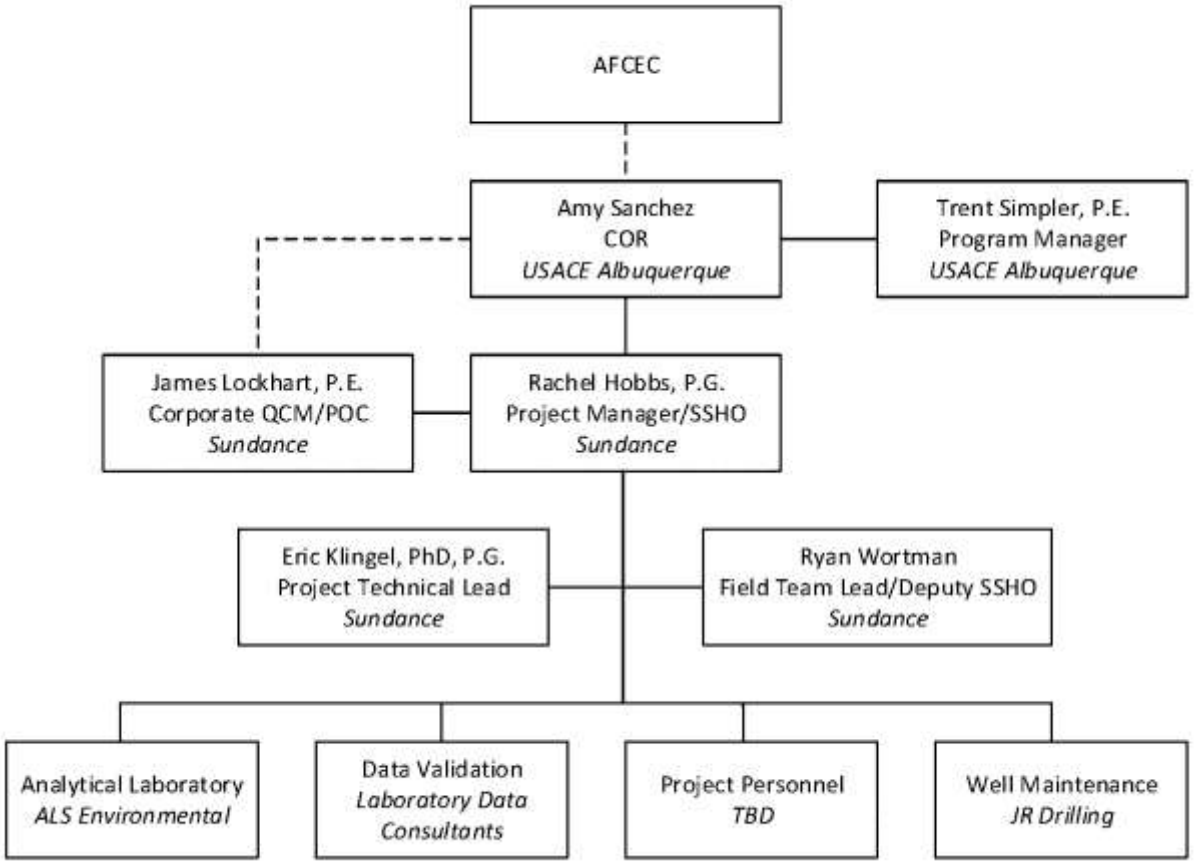
- Sample four drinking water supply wells monthly for EDB by EPA method 504.1, and benzene, toluene, ethylbenzene, and xylenes (BTEX) by EPA Method 524.2 (Figure 2-3).

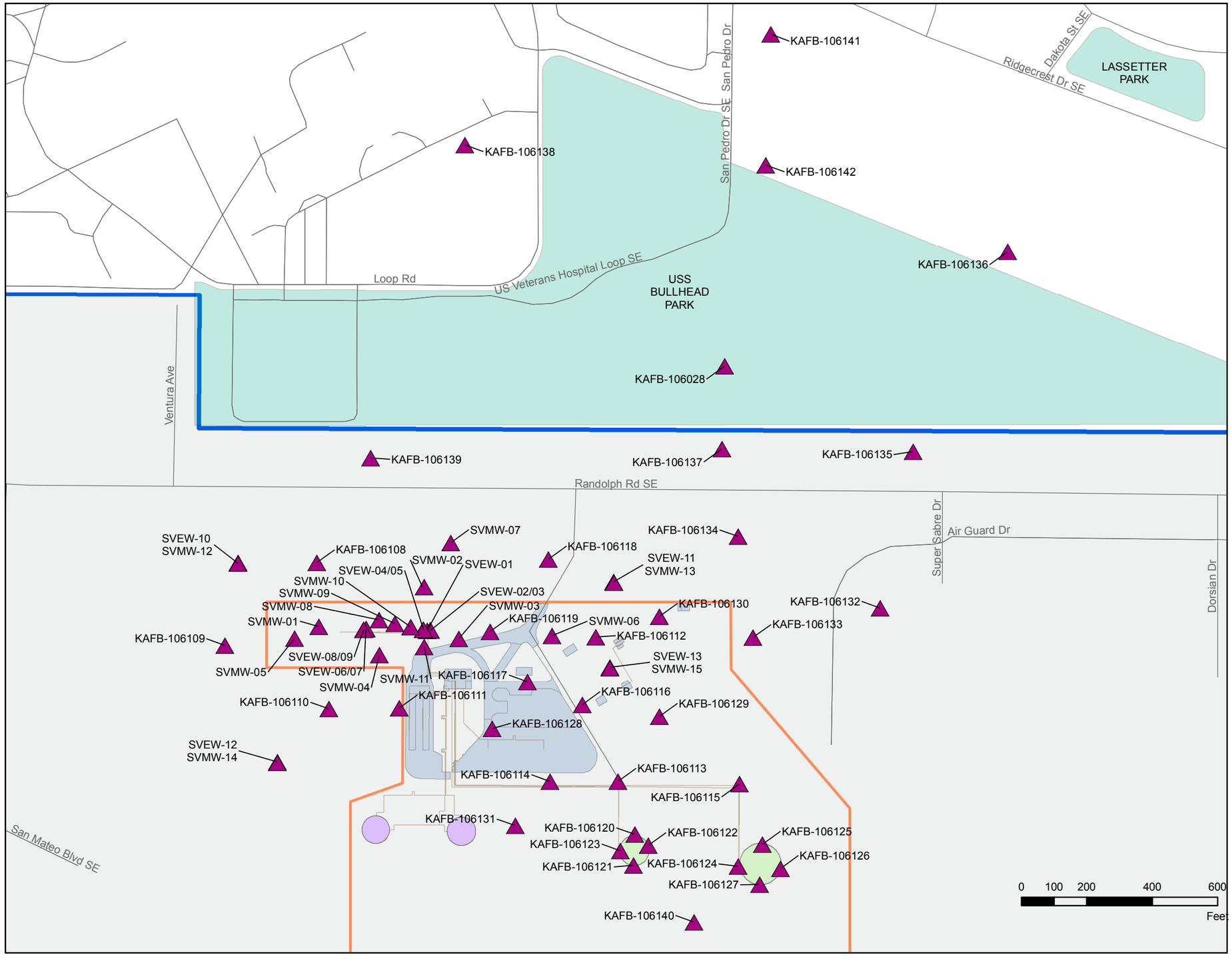
- Perform annual maintenance of the SVM network.

- Abandon and install SVMPs as necessary.

- Analyze soil vapor and drinking water supply well samples, and report results for soil vapor and drinking water supply wells in Quarterly Monitoring and Site Investigation Reports.

Figure 2-1. Quality Assurance Organization
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico





Legend

- Kirtland Air Force Base Installation Area
- City of Albuquerque Parks
- Roads
- Bulk Fuels Facility Area
- Soil Vapor Monitoring Location
- Bulk Fuels Facility Infrastructure
- Current Fuel Storage Tanks
- Former Fuel Storage Tanks
- Fuel Transfer Lines

N

Credits: City of Albuquerque

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

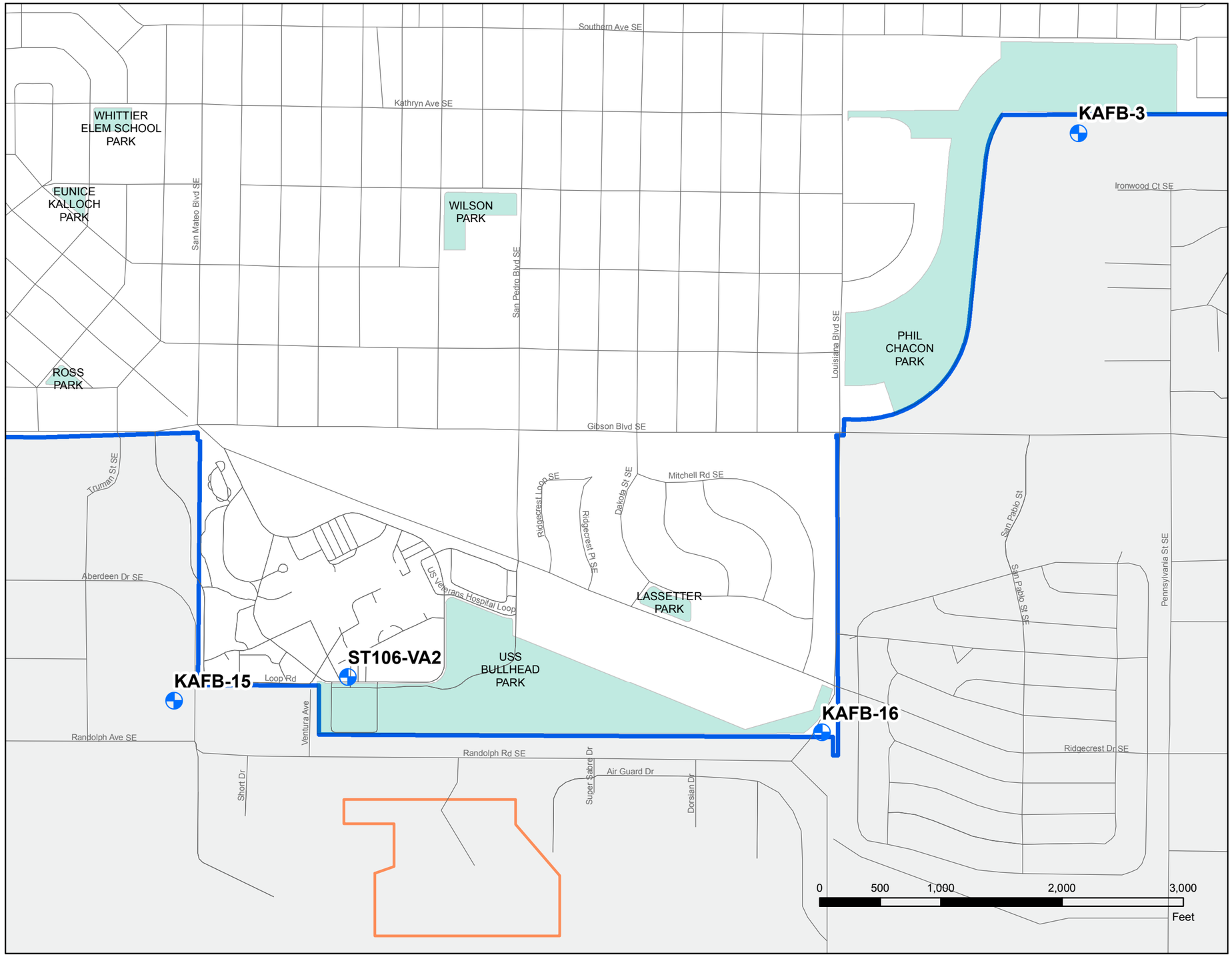
Figure 2-2

Soil Vapor Monitoring Locations
Quality Assurance Project Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016



Legend

- Kirtland Air Force Base Installation Area
- City of Albuquerque Parks
- Roads
- Bulk Fuels Facility Area
- Drinking Water Supply Well

N

Credits: City of Albuquerque

Coordinate System:
NAD 1927 StatePlane New Mexico Central FIPS 3002 Feet

Figure 2-3

Drinking Water Supply Wells
Quality Assurance Project Plan

Soil Vapor Monitoring
and Drinking Water Monitoring

Bulk Fuels Facility
Kirtland Air Force Base, New Mexico

Last Revised: 4/19/2016

Table 2-1. Personnel Qualifications
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Position/Staff	Qualifications	Responsibilities	Authority Level
Vice President of Operations: Jim Lockhart, P.E.	• BSME, MBA; • 33 years' experience in environmental remediation and engineering; • 25 years in management of environmental and engineering projects.	• As Sundance Officer, authorized to negotiate and commit resources; • Primary POC for USACE on contractual and programmatic items; • Ensures consistency in deliverables and cost/performance reporting and progress reporting/invoicing; • Coordinates issue resolution as needed with the COR and/or CO.	• Coordinates corrective action at programmatic level.
~~Project Manager:~~ ~~Patrick Scher, P.G.~~	• ~~M.S. in Geology;~~ • ~~Registered Professional Geologist in two states;~~ • ~~30 years' experience in environmental remediation/compliance;~~ • ~~25 years' experience in DoD Project and Program Management;~~ • ~~15 years' experience as technical lead/PM on complex environmental projects;~~ • ~~25-year accident free track record.~~	• ~~Ensures that all work is accomplished with adequate internal controls;~~ • ~~Main point of contact for USACE on project-specific matters;~~ • ~~Reviews/confirms technical approach from kickoff meeting and throughout project execution to ensure project objectives are met;~~ • ~~Assembles and schedules resources;~~ • ~~Ensures on-schedule and high-quality services are delivered within budget;~~ • ~~Manages subcontractors;~~ • ~~Coordinates Sundance's participation in the public meeting; community relations process;~~ • ~~Identifies and mitigates risks related to execution of the technical aspects of the work and ensures site safety;~~ • ~~Ensures work is performed in accordance with USACE/USAF Guidelines, state/federal regulations;~~ • ~~Applies lessons learned from current and past projects;~~ • ~~Responsible for front and back end transition activities to ensure continuity on the project;~~ • ~~Ensures public relations sensitivities are met.~~	• ~~Full responsibility and authority to execute Task Orders;~~ • ~~Approves subcontractor invoices, project charges, and deliverables;~~ • ~~Implements corrective action;~~ • ~~Stops work for any reason related to the project.~~

Table 2-1. Personnel Qualifications
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page ~~2 of 3~~ 2 of 4)

Position/Staff	Qualifications	Responsibilities	Authority Level
~~Project Geologist, Technical Lead/~~Project Manager/SSHO: Rachel Hobbs, P.G.	• M.S. in Geology; • Registered Professional Geologist in the state of Tennessee; • 5 years' experience in environmental remediation; • Past experience coordinating Kirtland BFF project tasks.	• Reports to the PM and serves as the Alternative PM; • Overall responsibility for design, implementation, and management of sampling activities; • Reviews all WP, reporting, and data deliverables; • Coordinates with Field Personnel for oversight and quality control; • Responsible for providing input for the design of the corrective actions and reviews corrective elements specific to sampling; • Oversees development of APP in accordance with Engineer Manual 385-1-1 and Occupational Safety and Health Administration regulations; • Assists PM and procurement staff in verification of safety performance of Sundance staff and subcontractors. Investigates any incidents, accidents, or safety violations Performs safety audits; • Manages monitoring reports.	• Approves APPs/SSHPs and all modifications before issuance to USACE; • Manages Health and Safety Program and directs training and required attendance; • Investigates safety concerns raised by staff; • Investigates any accidents; • Stops work for any reason including noncompliance/safety violation, or quality violations.
Project Technical Lead: Eric Klingel, PhD, P.G.	• PhD. in Geology; • Registered Professional Geologist in the state of North Carolina and South Carolina; • 30 years' experience in environmental site characterization, remediation and project management.	• Reports to the Project Manager and serves as the Alternative Project Manager; • Overall responsibility for design, implementation, and management of sampling activities; • Reviews all work plans, reporting, and data deliverables; • Coordinates with Field Personnel for oversight and QC; • Responsible for providing input for the design of the corrective actions and reviews corrective elements specific to sampling;	• Stops work for any reason including noncompliance/safety violation, or quality violations.

Table 2-1. Personnel Qualifications
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (~~Concluded, Page 3 of 3~~ Continued, Page 3 of 4)

		• Oversees development of APP in accordance with Engineer Manual 385-1-1 and Occupational Safety and Health Administration regulations; • Assists Project Manager and procurement staff in verification of safety performance of subcontractors Investigates any incidents, accidents, or safety violations Performs safety audits; Manages monitoring reports.	
Field Team Lead/Deputy SSHO: Ryan Wortman	• B.S. in Geology; 1+ year past experience coordinating Kirtland BFF project tasks.	• Reports to Technical Lead and/or PM; • Oversees sampling team and sampling activities; • Coordinates with the PM and Project Technical Lead on any deviations from the QAPjP due to changed field conditions such that data quality objectives are met; • Coordinates with SSHO to ensure that project activities are being performed in accordance with the APP. • Performs Health and Safety oversite in SSHO's absence.	Stop sampling work at any time due to safety or quality violations.

Acronyms & Abbreviations:
APP – Accident Prevention Plan
BFF – Bulk Fuels Facility
B.S. – Bachelor of Science Degree
BSME – Bachelor of Science in Mechanical Engineering

Table 2-1. Personnel Qualifications
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 4 of 4)

CO – Contracting Officer
COR – Contracting Officer's Representative
DoD – Department of Defense
MA – Master of Arts
MBA – Master of Business Administration
M.S. – Master of Science Degree
P.E. – Professional Engineer
P.G. – Professional Geologist
PhD – Doctor of Philosophy
PM – Project Manager
POC – point of contact
QAPjP – Quality Assurance Project Plan
SSHO – Site Safety and Health Officer
SSHP – Site Safety and Health Plan
USACE – United States Army Corps of Engineers
USAF – United States Air Force

3 DATA GENERATION AND ACQUISITION

3.1 Sampling Design

This section discusses the sampling and analysis strategy for drinking water and soil vapor samples required to meet the project data quality objectives (DQOs). SVM locations and drinking water supply wells are shown in Figures 2-2 and 2-3. Soil samples may be collected as part of the installation of soil vapor monitoring points. If soil sampling is performed as part of this project, the text, tables, and figures of this QAPjP will be revised to include necessary information.

Drinking water and soil vapor samples will be labeled, packaged, and shipped to the environmental Laboratory. Drinking water samples will be shipped to TestAmerica Laboratories in Savannah, Georgia. Soil vapor samples will be shipped to ALS Environmental Laboratory, Inc. (ALS) in Simi Valley, California. ALS and TestAmerica maintain U.S. Department of Defense (DoD) Environmental Laboratory Accreditation Programs (ELAP) certification for the analyses required under this contract. Laboratory Data Consultants, Inc. (LDC) will perform the third party data validation utilizing established data validation procedures (manually or automated) to perform 100 percent (%) review and EPA Level III data validation.

3.1.1 Soil Vapor Monitoring

There are 56 SVM locations, most of which are installed in nested SVM locations that contain multiple monitoring points for a total of 284 SVMPs (Figure 2-2). Based on information that will be collected throughout the implementation of this project, the network may be modified. New SVMPs may be added and some may be removed from the sampling program based on Vadose Zone Working Group recommendations and agreements with NMED. Soil vapor sampling will be performed in accordance with the Soil Vapor Monitoring and Drinking Water Monitoring Work Plan (WP), to which this QAPjP is attached (Appendix C).

Soil vapor samples will be analyzed for the following parameters:

- VOCs and total petroleum hydrocarbon (TPH) gasoline: EPA Method TO-15
- EDB: CARB 422
- APH (C5-C8 and C9-C12): Massachusetts Department of Environmental Protection (MA DEP)
- Fixed gases (hydrogen, oxygen [O_2], nitrogen, carbon monoxide, carbon dioxide, methane): E3C

3.1.2 Drinking Water Supply Well Monitoring

The drinking water supply well investigation activities at the BFF covered under this QAPjP are limited to four drinking water supply wells (KAFB-3, KAFB-15, KAFB-16, and ST106-VA2). These existing drinking water supply wells at KAFB and the Veteran's Administration (VA) Hospital area able to actively provide drinking water to the facilities' employees and inhabitants. All operational drinking water supply well locations will be sampled monthly, and analyzed for EDB using EPA Method 504.1, and for BTEX using EPA Method 524. Drinking water well sampling will be performed in accordance with the WP.

3.1.3 Soil Vapor Monitoring Well Installation, and Analysis

As part of the vadose zone sampling and analysis, one or more additional SVM locations, consisting of up to six SVMPs approximately 450 ft below ground surface (bgs), may need to be installed. Depending on the objectives of the borehole drilling and well installation, soil samples may be collected during borehole advancement. Soil sample collection techniques, analytical methods, and criteria for collection will be specified in the associated WP. Soil and subsequent soil vapor samples are collected for chemical analysis and will be shipped to ALS for analysis in accordance with DoD Quality Systems Manual (QSM) Version 5.0 (DoD 2013) and laboratory-specific standard operating procedures (SOPs).

3.1.4 Investigation-Derived Waste Management

It is not anticipated that any soil or water investigation-derived waste (IDW) will be generated during monitoring. SVM does not generate any containerized waste. In addition, any excess drinking water will be disposed of via KAFB's waste water treatment system or to the ground. Non-reusable personal protective equipment (PPE) will be disposed of in accordance with the project Accident Prevention Plan (APP) (Section 9.0). Any additional waste associated with sampling (plastic bags, paper waste, etcetera) will be collected and disposed of via the City of Albuquerque's (COA) waste management system.

IDW management details pertaining to any SVM well installation or abandonment that may be performed will be provided in future revisions of, or addendums to the WP. Once the drilling techniques and waste streams are determined, specifications will be submitted describing the management of that waste.

3.2 Quality Objectives and Criteria for Measurement Data

The DQO process is designed to ensure that the type, quantity, and quality of environmental data used for decision-making is appropriate for the intended application. The DQOs for the data collected in association with soil vapor and drinking water well monitoring includes the following:

- Support ongoing monitoring of the SVM network to evaluate soil vapor contamination in the vadose zone
- Support ongoing monitoring to ensure that dissolved phase EDB and BTEX have not impacted the existing drinking water wells

Soil vapor and drinking water well monitoring was initiated in the first quarter of calendar year (CY) 2016.

3.2.1 Comparison Criteria

Analytical methods selected for the project will provide sufficient sensitivity to meet the DQOs and NMED requirements, and will achieve the respective regulatory standard for all analytes in soil vapor, drinking water, and soil.

There are currently no applicable screening standards for soil vapor contamination used for the SWMU ST-106/SS-111 project.

Analytical results from the drinking water supply well monitoring events will be compared to EPA maximum contaminant levels (MCL) and New Mexico Water Quality Control Commission (NMWQCC) standards contained in New Mexico Administrative Code Title 20 – Environmental Protection, Chapter 6 – Water Quality, Part 2 – Ground and Surface Water Protection Section 20.6.2.3103.

Soil samples associated with SVM well installation or abandonment will be compared to EPA residential regional soil screening levels (RSL) (EPA, 2015) and NMED soil screening levels for residential receptors (NMED, 2015). Currently, there are no established regulatory standards for soil vapor. Regulatory limits are summarized in Attachment A with the laboratory analytical methods reporting limits.

Analytical methods, reporting limits, and screening criteria are presented in Attachment A, Tables A-1 and A-2. Analytical methods used by TestAmerica and ALS and reporting limits will provide sufficient sensitivity to meet the DQOs, EPA MCLs, and NMWQCC standards.

3.2.2 Project Performance and Acceptance Criteria

To limit uncertainty in obtained environmental data, criteria for the sensitivity, precision, bias, representativeness, completeness, and comparability (PARCC) parameters were developed and are presented in this QAPjP. Measurement errors will be controlled by using appropriate sampling and analytical methods, adhering to the DoD QSM (2013), following established SOPs, and having data review to verify laboratory processes. Field crews will be trained in appropriate sample collection procedures and will review the QAPjP before sample collection to limit sample collection errors. Subcontract analytical laboratories will have a copy of the QAPjP and will adhere to DoD QSM guidance to limit measurement errors. Following DoD QSM requirements, laboratories will conduct detection limit studies to verify method sensitivity. In addition, laboratories will perform limit of quantitation (LOQ) studies to verify precision and bias at the LOQ. For each matrix and each method, laboratories will analyze applicable QC samples, including laboratory method blanks, surrogates, laboratory control samples (LCS)/laboratory control sample duplicates (LCSD), matrix spike (MS)/matrix spike duplicates (MSD), and internal standards to determine that results of these QC samples are within acceptable precision and bias limits. Acceptance criteria for precision, bias, and sensitivity are presented in Attachment B. The data that meet these criteria will be of definitive quality and of less uncertainty than data which was acquired with a less rigorous approach.

3.3 Monitoring Methods

Soil vapor and drinking water monitoring will be performed in accordance with sampling methodologies presented in Sections 3.2 and 3.3 of the WP and the KAFB Basewide Plans (USAF, 2004).

3.3.1 Equipment Decontamination Procedure

The objective of field decontamination is to remove contaminants of concerns (CoC) from monitoring, and field equipment to concentrations that will not impact study objectives.

Decontamination procedures specific to SMV are outlined in Section 3.2.1.2 of the WP. It is not anticipated that any additional decontamination procedures will be required for soil vapor monitoring.

Decontamination procedures specific to drinking water supply well sampling are outlined in Section 3.3 Step 2 of the WP. It is not anticipated that any additional decontamination procedures will be required for drinking water supply well sampling.

3.4 Sample Handling and Custody

The following sections describe sample packaging and shipment, sample numbering and labeling, and chain-of-custody (COC) requirements associated with collecting soil vapor and drinking water samples.

3.4.1 Sample Packaging and Shipment

Soil vapor and drinking water samples will be collected in the appropriate certified clean sample containers provided by TestAmerica or ALS, and in accordance with the specific WP procedures and Table 3-1.

The primary objective of sample packaging and shipping requirements is to maintain sample integrity from the time a sample is collected until it is received at the analytical laboratory. Specific procedures for packaging and shipping of environmental samples are presented below:

Step 1. A sample label is attached to the sample bottle and completed with indelible ink;

Step 2. For water samples, a cooler will be used as a shipping container. In preparation for shipping samples, the drain plug will be taped shut so that no fluids, such as melted ice, will drain out of the cooler during shipment. A large plastic bag may be used as a liner for the cooler and packing material, such as bubble wrap, or Styrofoam beads, will be placed in the bottom of the liner. All water samples for chemical analysis must be shipped cooled to 4 degrees Celsius (°C) with ice. All samples will require icing prior to shipment. A temperature blank will be placed in every cooler shipment.

Step 3. Soil vapor samples will be returned to the lab in the soil vapor container boxes in which they were received. There are no temperature or preservative requirements for shipping of soil vapor samples.

Step 4. The soil vapor container liner will be taped closed, if used, and sufficient packing material will be used to prevent sample containers from making contact or rolling around during shipment.

Step 5. A copy of the COC form will be placed inside the sample cooler or box.

Step 6. The sample cooler or box will be closed and taped shut with packing tape.

Step 7. Custody seals will be placed on the sample cooler or box.

Step 8. The sample cooler or box will be shipped in accordance with the particular sample media and corresponding hold times.

3.4.2 Soil Vapor Monitoring Well and Drinking Water Supply Well Field Sample Identification

Field sample identification (ID) will be assigned consistent with the established KAFB sample ID nomenclature for soil vapor monitoring well and drinking water supply well field sample IDs. This will ensure that monitoring data associated with the BFF investigation will be recognizable and easily identified once uploaded to the USAF data repository.

3.4.2.1 Monitoring Well IDs

SVM well IDs will follow the format of the base designator (KAFB), the SWMU identifier (106) and the sequential monitoring well number (XXX). Well numbers will follow sequentially those wells that have already been installed at the BFF site.

3.4.2.2 Field Sample IDs

Sample IDs for soil vapor and drinking water samples will be assigned with a consistent and sequential sample number such that the laboratory will not be able to distinguish between samples of the same events. The designation for field samples will be as follows:

- Soil vapor – VA (last two digits of current year) then XXXX. Soil vapor samples collected in 2016 will be labeled VA16XXXX.
- Drinking water – GW (last two digits of current CY) then XXX. Drinking water supply well samples collected in 2016 will be labeled GW16XXX.

3.4.2.3 Field Quality Control Sample IDs

Field duplicate samples will have designations consistent with the sequential field sample IDs such that they will not be distinguishable by the laboratories as being a duplicate sample. Matrix spike (MS) and matrix spike duplicates (MSD) samples, trip blank, and field blank, samples will have sample designations as listed below:

- MS: GW16XXX-MS or -MSD
- Trip blanks (VOCs): GW16TB01, VA16TB01
- Field blanks/ambient blanks (VOCs): GW16AB01, VA16AB01

3.4.3 Sample Custody and Documentation

Sampling information will be recorded on a COC form and sample collection forms for tracking. All entries will be legible and recorded in indelible ink. Because samples may be analyzed at multiple laboratories, the terms laboratory and sample custodian are generic. The custody procedures described herein apply to all laboratories that are involved in the analysis of soil vapor, drinking water, and soil samples.

3.4.3.1 Chain of Custody Records

A blank example COC form is included in Appendix A of the WP. In addition to providing a custody exchange record for the samples, the COC serves as a formal request for sample analyses. The COC form will be completed and signed, thus becoming the COC record and distributed as follows:

- One copy retained by the sample coordinator for inclusion in the project files
- The original sent to the analytical laboratory with the sample shipment

After the laboratory receives field samples, the sample custodian will inventory each shipment before signing for it, and note on the original COC record any discrepancy in the number of samples, temperature of the cooler, or presence of broken samples. The Sundance PM and/or technical lead will be notified immediately of any problems identified with shipped samples, and will determine the appropriate course of action and if project budget or schedule may be impacted.

The laboratory will initiate an internal COC that will track the sample within the various areas of the laboratory. The relinquishing signature of the sample custodian and the custody acceptance signature of the laboratory personnel document that custody of the sample has been transferred appropriately. This procedure will be followed each time a sample changes hands. The laboratory will archive the samples

and maintain them in custody as required by the contract or until further notification from Sundance, at which time the samples will either be returned to the project for disposal, or disposed of by the laboratory.

3.4.3.2 Field Sample Custody

The COC form or record will be the controlling document to ensure that sample custody is maintained. Upon collecting a sample, sampling personnel will initiate the COC in the field. Each individual who has the sample(s) in their possession will sign the COC. Each time the sample custody is transferred, the former custodian will sign the COC on the "Relinquished by" line, and the new custodian will sign the COC on the "Received by" line. The date, time, and name of their project or company affiliation will accompany each signature.

The waybill number or courier name will be recorded on the COC form when a commercial carrier is used. The shipping container will be secured with two custody seals, thereby allowing shipping personnel to maintain custody until receipt by the laboratory.

If the laboratory sample custodian judges sample custody to be invalid (e.g., custody seals have been broken), the laboratory will notify the Sundance PM and/or technical lead who will in turn contact the field team to resolve any discrepancies with field sample documentation. Any corrections required to be made to COC forms will be made by the field team, reviewed by the Sundance PM and/or technical lead to determine impact to sample custody, and transferred to the laboratory. Sample receipt discrepancies will be noted by the laboratory upon sample login.

3.4.3.3 Sample Collection Log

The Sample Collection Log form will be used to document all samples collected in the field. A copy of this form for soil vapor and drinking water can be found in Appendix A of the WP. All entries will be recorded in indelible ink. The sample team will cross out any unused portions and sign each page.

3.4.3.4 Vapor Purge Log

The Vapor Purge Log form will be used to document field sample collection information associated with SVM. A copy of this form can be found in Appendix A of the WP. All entries will be recorded in indelible ink, and will be reviewed by the sampling team. At a minimum, the vapor purge log will contain the following information:

- Project name and site
- SVMP identification number
- Field team /personnel name
- Sample date and time
- Weather conditions
- SVMP observations
- Purge calculations
- Purge volume
- Field measurements (carbon dioxide, O_2, and hydrocarbons [HC])

The Vapor Purge Log will undergo an independent QC review by a field team member other than the author or designee before shipping the samples to the offsite laboratory.

3.4.3.5 Document Corrections

Changes or corrections to any project, field, or analytical documentation will be made by crossing out the item with a single line, initialing by the person performing the correction, and dating the correction. The original item, although erroneous, will remain legible beneath the cross out. The new information will be written above the crossed-out item. Corrections will be written clearly and legibly with indelible ink.

3.5 Analytical Methods

Analytical methods, container, and preservative requirements for soil vapor and drinking water samples are summarized in Tables 3-2 and 3-3. The required target analytes for each method, applicable regulatory limits, project reporting limits, and laboratory LOQs are presented in Attachments A and B.

3.6 Quality Control

This section discusses field and laboratory QC requirements.

3.6.1 Field Quality Control Samples

Field QC samples will be collected and analyzed during the project to assess the precision and accuracy of the sampling program. Field QC samples for this project will include MS/MSD samples, field duplicates, trip blanks for VOC samples, and temperature blanks, and QA split samples if requested by USACE and NMED as discussed below.

3.6.1.1 Matrix Spike and Matrix-Spike Duplicate

MS/MSD samples will be collected at one pair per 20 drinking water samples; at least one per sampling event. MS/MSD analyses will not be performed on soil vapor samples as MS/MSD analysis for these methods and matrix are not applicable. Accuracy for these analyses will be assessed through a review of field duplicates, laboratory duplicates, and surrogate recoveries (when applicable). Field personnel will collect extra volumes for water for MS/MSD analysis and designate the MS/MSD sample(s) on the COC record (Appendix A of the WP).

3.6.1.2 Field Duplicates

Field duplicate pairs consist of two samples of the same matrix (a primary and a duplicate) collected at the same time and location to the extent possible, using the same sampling techniques. The purpose of field duplicate samples is to evaluate sampling precision. Field duplicate samples will be collected for soil vapor monitoring and drinking water sampling. Field duplicate samples will be collected at a frequency of 10% and will be analyzed for the same analytical parameters as their corresponding primary samples. For this project, the acceptance criteria for field duplicate precision is established at less than or equal to 35% for drinking water samples, and 50% for soil vapor samples. Field duplicate precision will be calculated when target analytes are detected above the reporting limit in both the primary and duplicate sample.

No field duplicates will be collected for IDW characterization purposes.

3.6.1.3 Performance Evaluation Samples

Use and anaylsis of performance evaluation samples will be implemented by the client or designee if deemed necessary. Performance evaluation samples are independent clean matrix samples that are spiked with project-specific target compounds and introduced into the sampling program by the field team.

Performance evaluation samples are then submitted to the project laboratory for analysis as blind samples to be evaluated by the USAF upon receipt of data deliverables. These results may serve as an independent QA check for the field sampling and analytical method protocol precision.

3.6.1.4 Trip Blanks

Trip blank samples will accompany each shipment containing soil vapor, drinking water and soil samples for VOC analysis. Trip blanks for drinking water samples will be 40-milliliter (mL) volatile organic analysis vials that contain analyte-free water, which are kept with the field samples during sampling and shipment to an offsite laboratory. Trip blanks for soil vapor samples are 1-liter Bottle-Vacs that are kept with field samples during soil vapor sampling and shipment to an offsite laboratory. The vacuum of the Bottle-Vac will be recorded, but the valve will not be opened, and the container will be returned to the lab with the shipment of soil vapor samples. Results of trip blank samples will be used to determine if samples have been contaminated with VOCs during sampling or shipment to the laboratory.

3.6.1.5 Temperature Blanks

Each cooler containing drinking water samples will be shipped with a temperature blank. A temperature blank is a sample container filled with tap water and shipped in the cooler to the offsite laboratory. The laboratory will record the temperature of the blank upon receipt of the samples. The temperature blank is to ensure that the temperature of the samples when received at the laboratory is less than or equal to 4°C. Temperature blanks are not required to accompany soil vapor samples to the offsite laboratory.

3.6.2 Laboratory Quality Control Samples

To ensure acceptable data quality, laboratory QC analysis will be performed for each method and for each matrix. Laboratory QC samples will include method blanks, initial and continuing calibration blanks, surrogates, LCSs, and internal standards. Tables 3-4 and 3-5 present these QC samples, acceptance criteria, and corrective actions. These QC requirements are consistent with the DoD QSM (2013) guidance. The DoD QSM and laboratory in-house control limits are presented in Attachment B.

3.7 Instrument/Equipment Testing, Inspection, and Maintenance

Field and analytical instrument testing, inspection, and maintenance requirements are described in this section. All requirements are presented in tabular format on Table 3-6 (Field Instrument Quality Control), Table 3-7 (Laboratory Instrument Quality Control – Drinking Water Monitoring), and Table 3-8 (Laboratory Instrument Quality Control – Soil Vapor Monitoring), and in Attachment B.

Other activities such as well installation and abandonment may be performed as part of this project. When designs for these activities are finalized, any additional field measurement specifications for soil boring logs, well reports, and surveying will be included in a subsequent revision to this QAPjP.

3.8 Laboratory Instrument/Equipment Calibration and Frequency

Laboratory instrument calibration requirements, frequencies, and corrective actions for each method in this section. These calibration requirements are established in accordance with the DoD QSM requirements. Calibration is a reproducible reference point to which all sample measurements can be correlated. Instrumentation calibration is necessary for accurate sample quantitation. Calibrations establish the dynamic range of an instrument, establish response factors to be used for quantitation, and demonstrate instrument sensitivity.

All calibration requirements are presented in tabular format in Table 3-9 (Analytical Instrument Calibration - Gas Chromatography/Mass Spectrometry), and Table 3-10 (Analytical Instrument Calibration (Gas Chromatography), and Attachment B.

3.9 Inspection/Acceptance of Supplies and Consumables

The accuracy of sample target analyte quantitation is directly related to the accuracy of the standards used for instrument calibration. To ensure the highest quality standard, primary reference standards used by laboratories are obtained from reliable commercial sources. Inorganic standards must be traceable to the National Institute of Standards and Technology (NIST); organic standards must be traceable to NIST or American Association of Laboratory Accreditation vendors when available. When standards are received at the laboratory, the date received, supplier, lot number, purity and concentration, and expiration date are recorded in a standard preparation log book. Vendor certifications sent with the standards are also filed and are available upon request.

Standards purchased by the laboratory may be in a pure form, in a stock, or in a working standard solution. All standards made are given a standard identification number and have the following information recorded in a standards log book: source of standard used to prepare dilution; preparer's initials; initial concentration; final concentration; solvent; source and lot number of solvent; volume of final solution; and volume of standard diluted. Records must unambiguously trace the preparation of standards, their use in calibration, and the quantitation of sample results. After preparation and before routine use, the identity and concentration of standards are verified. Verification procedures include a check for chromatographic purity (if applicable) and verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source. Reagents are also examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used. Standards are routinely checked for signs of deterioration (e.g., discoloration, formation of precipitates, or changes in concentration) and are discarded if deterioration is suspected or their expiration date has passed. Expiration dates may be taken from the vendor recommendation, the analytical methods, or from internal research.

Table 3-1. Sample Requirements for Analytical Testing
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Drinking Water and Soil Vapor Samples					
Matrix	**Parameter[1]**	**Container[2,3]**	**Preservation**	**Maximum Holding Times[4]**	
				Extraction	**Analysis**
Water	BTEX	3 x 40-mL G, Septa Vial	Ice to 4°C 4 drops conc. HCl to pH<2	---	14 days
Water	EDB	3 x 40-mL G, Septa Vial	Ice to 4°C	---	14 days
Vapor	VOCs/APH	1 x 1-L Bottle Vac	None	N/A	28 days
Vapor	Fixed gases	1 x 1-L Bottle Vac	None	N/A	30 days
Vapor	CARB 422	1 x 1-L Bottle Vac	None	N/A	30 days

Acronyms and Abbreviations:
< = less than
°C = degrees Celsius; APH = air-phase petroleum hydrocarbon
BTEX = benzene, toluene, ethylbenzene, and xylenes
CARB = California Air Resources Board
EDB = ethylene dibromide
G = glass
HCl = hydrochloric acid
L = liter
mL = milliliter
pH = potential hydrogen
VOC = volatile organic compound

1. All containers must have Teflon-lined seals.
2. (Teflon-lined septa for volatile organic analysis [VOA] vials).
3. Sample preservation will be completed in the field immediately upon sample collection.
4. When only one holding time is given, it implies total holding time from sampling until analysis.

Table 3-2. Analytical Method, Preservation, and Holding Time Requirements - Drinking Water Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	Analytical Group	Analytical and Preparation Method/SOP Reference	Sample Volume	Container (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Water	BTEX – EPA 524.2	Preparation: EPA 524.2 Analysis: EPA 524.2	40 mL	3 X 40 mL VOA with Teflon® septa	HCL to pH <2 Cool at 0-4°C	14 days for analysis
Water	EDB – EPA 504.1	Preparation: EPA 504.1 Analysis: EPA 504.1	40 mL	3 X 40 mL VOA with Teflon® septa	Cool at 0-4°C	14 days for analysis

Acronyms and Abbreviations:
< = less than
°C = degrees Celsius
BTEX = benzene, toluene, ethylbenzenes, and xylenes
EDB = ethylene dibromide
EPA = United States Environmental Protection Agency
HCl = hydrochloric acid
mL = milliliter
NA = not applicable
pH = potential hydrogen
SOP = standard operating procedure
SVOC = semi volatile organic compound
VOA = volatile organic analysis

Table 3-3. Analytical Method, Preservation, and Holding Time Requirements – Soil Vapor Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	Analytical Group	Analytical and Preparation Method/SOP Reference	Sample Volume	Container (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Vapor	VOCs EPA TO15	Preparation: EPA TO15 Analysis: EPA TO15	1 L	1 L Bottle Vac Canister; 1L for Vapor	NA	30 days for analysis
Vapor	APH – Method MA DEP	Preparation: Method MA DEP Analysis: Method MA DEP	1 L	1 L Bottle Vac Canister	NA	28 days for analysis
Vapor	Fixed Gases – ASTM D2504	Preparation: ASTM D2504 Analysis: ASTM D2504	1 L	1 L Bottle Vac Canister	NA	30 days for analysis
Vapor	CARB 422	Preparation: CARB 422 Analysis: CARB 422	1 L	1 L Bottle Vac Canister	NA	30 days for analysis

Acronyms & Abbreviations:
APH = air-phase petroleum hydrocarbon
ASTM = ASTM International
CARB = California Air Resources Board
EPA = U.S. Environmental Protection Agency
L = liter
MA DEP = Massachusetts Department of Environmental Protection
NA = not applicable
SOP = standard operating procedure
TPH = total petroleum hydrocarbon
VOC = volatile organic compound

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**VOCs, BTEX, and APH**					
Analytical Method	**Methods 524.2, MA DEP, and TO15**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicators**	**Measurement Performance Criteria**
Internal standards	Every field sample and QC samples	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Correct problem, then re- reanalyze affected samples.	Lab Manager/Analyst	Bias	RT within ±30 seconds and area count within -50% to +100%
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/Analyst	Representativeness	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 2 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**VOCs, BTEX, and APH**					
Analytical Method	**EPA Methods 524.2, MA DEP, and TO15**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicators**	**Measurement Performance Criteria**
MS/MSD for all analytes	One MS/MSD pair per preparation batch per matrix *Not performed on vapor samples	EPA 524.2 and 504.1, MA DEP: LCS control limits specified by laboratory SOP	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/Analyst	Precisions and Bias	EPA 524.2 and 504.1, MA DEP: LCS control limits specified by laboratory SOP
LCS or LCS/LCSD pair for all analytes	One LCS or LCS/LCSD pair per preparation batch per matrix	EPA 524.2 and 504.1, MA DEP: LCS control limits specified by laboratory SOP TO15: LCS control limits specified in the DOD QSM	Correct problem, then re-reanalyze the LCS and all associated batch samples	Lab Manager/Analyst	Precisions and Bias	EPA 524.2 and 504.1, MA DEP: LCS control limits specified by laboratory SOP TO15: LCS control limits specified in the DOD QSM

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 3 of 5)

Matrix	Drinking Water and Soil Vapor					
Analytical Group	VOCs, BTEX, and APH					
Analytical Method	EPA Methods 524.2, MA DEP, and TO15					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicators**	**Measurement Performance Criteria**
Surrogate standards	Every field sample and QC sample	EPA 524.2 and 504.1: Surrogate recovery acceptance criteria specified in laboratory SOP TO15: Specified in DOD QSM	Correct problem, then re-reanalyze all affected samples	Lab Manager/Analyst	Bias	EPA 524.2 and 504.1: Surrogate recovery acceptance criteria specified in laboratory SOP TO15: Specified in DOD QSM Version 5.0
Sample duplicate	Every 20 samples	TO15: Specified in DOD QSM MA DEP: Surrogate recovery acceptance criteria specified in laboratory SOP	NA	Lab Manager/Analyst	Bias	TO15: Specified in DOD QSM MA DEP: Surrogate recovery acceptance criteria specified in laboratory SOP
MDL study	Initial setup *Not run for MA APH	Detection limits established will be below the LOQs	Correct problem, then repeat the MDL study	Lab Manager/Analyst	Sensitivity	

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 4 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**VOCs, BTEX, and APH**					
Analytical Method	**EPA Methods 524.2, MA DEP, and TO15**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
LOD study	Initial setup and quarterly LOD verification *Not run for MA APH	Signal to noise ratio at the LOD will be greater than 3 and meet method requirements.	Correct problem, then repeat detection limit study and LOD verification at a higher concentration, or pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration in accordance with DoD QSM requirements.	Lab Manager/Analyst	Sensitivity	
LOQ study	Annually and quarterly LOQ verification	LOQ will be greater than LOD and within calibration range. Laboratory procedure for establishing the LOQ will empirically demonstrate precision and bias at the LOQ LOQ>LOD>DL		Lab Manager/Analyst	Sensitivity	

Table 3-4. Laboratory QC Samples - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 5 of 5)

Acronyms and Abbreviations:
% = percent
APH = air-phase petroleum hydrocarbon
DL = detection limit
DoD = U.S. Department of Defense
EPA = U.S. Environmental Protection Agency
LCS = laboratory control sample
LCSD =laboratory control sample duplicate
LOD = limit of detection
LOQ = limit of quantitation
MA APH = Massachusetts Air-Phase Petroleum Hydrocarbon
MA DEP = Massachusetts Department of Environmental Protection
MDL = method detection limit
MS = matrix spike
MSD = matrix spike duplicate
NA = not applicable
QC = quality control
QSM = Quality Systems Manual
RL = reporting limit
RPD = relative percent difference
RT = retention time
SVOC = semi volatile organic compound
VOC = volatile organic compound

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**EDB, TPH, Fixed Gases**					
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and >1/10 amount detected in project samples or 1/10 the regulatory limit (whichever is greater)	Correct problem, then re-extract and reanalyze method blank and all samples processed with the contaminated blank	Lab Manager/Analyst	Representativeness	No target analytes detected greater than one-half RL and >1/10 amount detected in project samples or 1/10 the regulatory limit (whichever is greater)
MS/MSD for all analytes	One MS/MSD pair per preparation batch per matrix *Not performed on vapor samples	EPA 504.1, MA DEP: Laboratory in-house LCS control limits RPD less than 30% between MS and MSD	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples	Lab Manager/Analyst	Precisions and Bias	EPA 504.1, MA DEP: Laboratory in-house LCS control limits RPD less than 30% between MS and MSD

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 2 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**EDB, TPH, Fixed Gases**					
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
LCS or LCS/LCSD pair for all analytes	One LCS or LCS/LCSD pair per preparation batch per matrix	EPA 524.2 and 504.1, MA-DEP: Laboratory in-house LCS control limits	Correct problem, then re-extract and reanalyze the LCS and all associated batch samples	Lab Manager/Analyst	Precisions and Bias	EPA 524.2 and 504.1, MA-DEP: Laboratory in-house LCS control
Surrogate standards	Every field sample and QC sample *Not added to CARB422 or fixed gasses	EPA 524.2 and 504.1, MA-DEP: Laboratory in-house surrogate acceptance criteria	Correct problem, then re-extract and reanalyze all affected samples	Lab Manager/Analyst	Bias	EPA 524.2 and 504.1, MA-DEP: Laboratory in-house surrogate acceptance criteria

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 3 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**EDB, TPH, Fixed Gases**					
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
Confirmation of positive results using second column or second detector	All positive results must be confirmed	Same calibration and QC requirements as for initial or primary column analysis. RPD between primary and second column results less than 40%	NA	Lab Manager/Analyst	Precision	RPD between primary and second column results less than 40%
MDL study	Initial setup *Not run for fixed gasses	Detection limits established will be below the LOQs	Correct problem, then repeat the MDL study in accordance with DoD QSM requirements	Lab Manager/Analyst	Sensitivity	

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 4 of 5)

Matrix	**Drinking Water and Soil Vapor**					
Analytical Group	**EDB, TPH, Fixed Gases**					
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**					
QC Sample	**Frequency**	**QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**	**Data Quality Indicator**	**Measurement Performance Criteria**
LOD study	Initial setup and quarterly LOD verification *Not run for CARB422 or fixed gasses	Signal to noise ratio at the LOD will be greater than 3 and meet method requirements.	Correct problem, then repeat detection limit study and LOD verification at a higher concentration, or pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration per DoD QSM	Lab Manager/Analyst	Sensitivity	
LOQ study	Annually and quarterly LOQ verification	LOQ will be greater than LOD and within calibration range. Laboratory procedure for establishing the LOQ will empirically demonstrate precision and bias at the LOQ LOQ>LOD>DL		Lab Manager/Analyst	Sensitivity	

Table 3-5. Laboratory QC Samples - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 5 of 5)

Acronyms and Abbreviations:
% = percent
ASTM = ASTM International
CARB=California Air Resources Board
DoD = U.S. Department of Defense
EDB = ethylene dibromide
EPA = U.S. Environmental Protection Agency
EPH = extractable petroleum hydrocarbon
LCS = laboratory control sample
LCSD =laboratory control sample duplicate
LOD = limit of detection
LOQ = limit of quantitation
MA DEP = Massachusetts Department of Environmental Protection
MDL = method detection limit
MS = matrix spike
MSD = matrix spike duplicate
NA = not applicable
QC = quality control
QSM = Quality Systems Manual
RL = reporting limit
RPD = relative percent difference
SOP = standard operating procedure
TPH = total petroleum hydrocarbon
VPH = volatile petroleum hydrocarbon

Table 3-6. Field Instrument Quality Control
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Field Equipment	Calibration Verification Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Photoionization Detector	Check calibration to 100 parts per million isobutylene	Once per day before first use	± 5% of standard value	Recalibrate	Sundance Field Personnel	Manufacturer's Operation Manual
Horiba	Check calibration for petroleum hydrocarbons and CO_2 against a calibration standard of known concentrations in a premixed gas cylinder	Once per day before first use	± 10% of standard value	Recalibrate	Sundance Field Personnel	Manufacturer's Operation Manual – modified per WP
	Check calibration for O_2 against atmospheric concentrations	Once per day before first use	O_2>22%	Recalibrate	Sundance Field Personnel	Manufacturer's Operation Manual – modified per WP
YSI 556 Multi-Probe System Water Quality Meter	Check calibration for multi-probe meters against manufacturer provided calibration standards.	Once per day before first use	Manufacturer's Standard	Recalibrate	Sundance Field Personnel	Manufacturer's Operation Manual – modified per WP

Acronyms and Abbreviation
> = greater than
% = percent
CO_2 = carbon dioxide
O_2 = oxygen
SOP = standard operating procedure
Sundance = Sundance Consulting, Inc.
WP = Work Plan
YSI = Yellow Springs Instruments

Table 3-7. Laboratory Instrument Quality Control – Drinking Water Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed.	Water samples	Ion source, injector liner, column, column flow, purge lines, purge flow, trap	Prior to initial calibration and/or as necessary	Acceptable tune and calibration or CCV	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	TestAmerica Analyst and Laboratory Manager	TestAmerica SA-VO-002, Rev. 5
MS	Change the injection port liner, column ferrule, and autosampler syringe as needed. Liners should be changed when recent sample analyses predict a problem with chromatographic performance. The autosampler should be cleaned periodically. This includes turret cleaning and cleaning or replacing the syringe.	Water Samples	injection port liner, column ferrule, and autosampler syringe	Prior to initial calibration and/or as necessary	Acceptable tune and calibration or CCV	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	TestAmerica Analyst and Laboratory Manager	TestAmerica SA-SG-060, Rev. 12

Table 3-7. Laboratory Instrument Quality Control – Drinking Water Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 2 of 2)

Acronyms and Abbreviations:
BFB = bromofluorobenzene
CCV = continuing calibration verification
GC/MS = gas chromatography/mass spectrometry
SOP = standard operating procedure
VOC = volatile organic compound

Table 3-8. Laboratory Instrument Quality Control – Soil Vapor Monitoring
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Daily/regular as specified	Air samples	Instrument operating parameters	Daily	Per SOP	Recalibrate/ stop for service on failure	ALS Laboratory Analyst and Laboratory Manager	VOA-TO-15 Rev 22 VOA-MAPH Rev 9
GC	Daily during use.	Air/gas samples	Instrument operating parameters	Daily	Per SOP	Recalibrate/ stop for service on failure	ALS Laboratory Analyst and Laboratory Manager	VOA-EPA 3C Rev 13 SVO-CARB422 Rev 5

Acronyms and Abbreviations:
ALS = ALS Environmental Laboratory, Inc.
CARB = California Air Resources Board
EPA = U.S. Environmental Protection Agency
GC = gas chromatography
GC = gas chromatography
MS = mass spectrometry
SOP = standard operating procedure
VOA = volatile organic compound

Table 3-9. Analytical Instrument Calibration - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	**Drinking Water and Soil Vapor**				
Analytical Group	**VOCs, BTEX, and APH**				
Analytical Method	**EPA Methods 524.2, MA DEP, and TO15**				
Instrument	**Calibration Procedure**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**
GC/MS	Check of mass spectral ion intensities (tuning procedure) using bromofluorobenzene	Prior to initial calibration and meet frequency requirements specified in the method	Must meet the method requirements before samples are analyzed	Retune instrument and verify the tune acceptability, rerun the affected samples	Lab Manager/Analyst
	Five-point initial calibration for target analytes, lowest calibration standard at or near the LOQ in accordance with DoD QSM requirements	Initial calibration prior to sample analysis	TO15 and MA DEP: RSD is less than 30%per method requirements 524.2: RSD is less than 20% per method requirements	Correct problem, then rerun initial calibration in accordance with DoD QSM/method requirements	Lab Manager/Analyst

Table 3-9. Analytical Instrument Calibration - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 2 of 3)

Matrix	**Drinking Water and Soil Vapor**				
Analytical Group	**VOCs, BTEX, and APH**				
Analytical Method	**EPA Methods 524.2, MA DEP, and TO15**				
Instrument	**Calibration Procedure**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**
GC/MS	Second-source calibration verification in accordance with DoD QSM requirements	Once per five-point initial calibration	EPA 524.2: Less than 30% difference for all target analytes in accordance with method requirements. MA DEP: Less than 25% difference for all target analytes in accordance with method requirements	Correct problem, then rerun second source calibration verification in accordance with DoD QSM/method requirements	Lab Manager/Analyst
	Daily calibration verification in accordance with DoD QSM requirements	Before sample analysis and every 12 hours of analysis	EPA 524.2 and TO15: Less than 30% difference for all target analytes in accordance with method requirements MA DEP: Less than 25% difference for all target analytes per method requirements.	Correct problem, then rerun calibration verification in accordance with DoD QSM/method requirements	Lab Manager/Analyst
	Breakdown check	Before sample analysis and every 12 hours of analysis		Correct problem, then rerun breakdown check	Lab Manager/Analyst

Table 3-9. Analytical Instrument Calibration - Gas Chromatography/Mass Spectrometry
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 3 of 3)

Acronyms and Abbreviations:
% = percent
APH = air phase petroleum hydrocarbon
DDT = dichlorodipheyl trichloroethane
DoD = U.S. Department of Defense
EPA = U.S. Environmental Protection Agency
GC/MS = gas chromatography/mass spectrometry
LCS = laboratory control sample
LOQ = limit of quantitation
MA APH = Massachusetts Air-Phase Petroleum Hydrocarbon
MA DEP = Massachusetts Department of Environmental Protection
QSM = Quality Systems Manual
RSD = relative standard deviation
SVOC = semi volatile organic compound
VOC = volatile organic compound

Table 3-10. Analytical Instrument Calibration- Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Matrix	**Drinking Water and Soil Vapor**				
Analytical Group	**EDB, TPH, Fixed Gases**				
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**				
Instrument	**Calibration Procedure**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**
GC	Minimum five-point initial calibration for target analytes, lowest calibration standard at or near the LOQ in accordance with DoD QSM requirements Stable Isotope: perform external calibration of working standard per laboratory SOPs	Initial calibration prior to sample analysis	EPA 504.1: RSD less than or equal to 20% for all target analytes in accordance with DoD QSM requirements MA DEP: RSD less than 25% for all target analytes per method requirements	Correct problem, then rerun initial calibration in accordance with DoD QSM requirements.	Lab Manager/Analyst
	Second-source calibration verification	Once per five-point initial calibration	EPA 504.1: Less than 20% of expected values from the initial calibration for all target analytes in accordance with DoD QSM requirements MA DEP: Less than 25% of expected values from the initial calibration for all target analytes per method requirements	Correct problem, then rerun second source calibration verification in accordance with DoD QSM requirements.	Lab Manager/Analyst

Table 3-10. Analytical Instrument Calibration - Gas Chromatography
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 2 of 2)

Matrix	**Drinking Water and Soil Vapor**				
Analytical Group	**EDB, TPH, Fixed Gases**				
Analytical Method	**EPA Method 504.1, MA DEP, CARB422**				
Instrument	**Calibration Procedure**	**Frequency**	**Acceptance Criteria**	**Corrective Action**	**Person(s) Responsible for Corrective Actions**
GC	Daily calibration verification.	EPA 524.2 and 504.1, MA DEP, Before sample analysis and at frequency specified in the method	MA DEP: Less than 25% of expected values from the initial calibration for all target analytes per method requirements	Correct problem, then rerun calibration verification in accordance with DoD QSM requirements	Lab Manager/ Analyst

Acronyms and Abbreviations:
% = percent
ASTM = ASTM International
DoD = U.S. Department of Defense
EDB = ethylene dibromide
EPA = U.S. Environmental Protection Agency
EPH = extractable petroleum hydrocarbon
GC = gas chromatography
LOQ = limit of quantitation
MA DEP = Massachusetts Department of Environmental Protection
QSM = Quality Systems Manual
RSD = relative standard deviation
SOP = standard operating procedure
TPH = total petroleum hydrocarbon
VPH = volatile petroleum hydrocarbon

4 DATA VALIDATION AND USABILITY

4.1 Analytical Data Review, Verification, and Validation

The laboratory analyst who generates the analytical data will have primary responsibility for the correctness and completeness of data. Each step of this verification and review process will involve the evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of data is essential in ensuring that data of known quality is consistently generated. All data generated and reduced will follow well-documented in-house protocols.

4.1.1 Level 1: Technical or Peer Data Review

Analysts will review the quality of their work based on an established set of guidelines, including the QC criteria established in each method, in this QAPjP, and as stated within the laboratory QA manual (Attachment C). This review will, at a minimum, ensure that the following conditions have been met:

- Sample preparation information is correct and complete.
- Analysis information is correct and complete.
- Appropriate SOPs have been followed.
- Calculations are verified.
- There are no data transposition errors.
- Analytical values are correct and complete.
- QC samples results are within established control limits.
- Blank results are within appropriate QC limits.
- LCS results are within appropriate QC limits.
- Special sample preparation and analytical requirements have been met.
- Documentation is complete; for example, any anomalies and holding times have been documented and forms have been completed.

4.1.2 Level 2: Technical Data Review

A supervisor or data review specialist whose function is to provide an independent review of data packages will perform this review. This review will also be conducted according to an established set of guidelines and will be structured to verify the Level 1 data review. This review will, at a minimum, ensure that the following conditions have been met:

- Appropriate laboratory SOPs are followed.
- Calibration data are scientifically sound and appropriate to the method.
- QC samples results are within established guidelines.
- Qualitative identification of contaminants is correct.
- Manual integrations are justified and documented.
- Quantitative results and calculations are correct.
- Data is qualified correctly.

- Documentation is complete.
- The data package is complete and complies with contract requirements.

The Level 2 review will be structured so that all calibration data and QC sample results are reviewed and all of the analytical results from at least 10% of the samples are checked back to the sample preparation and analytical bench sheets. If no problems are found with the data package, the review will be considered complete. If discrepancies are identified, additional data evaluation is required.

4.1.3 Level 3: Administrative Quality Assurance Data Review

The laboratory QA Manager will review 10% of all data packages. This review should be similar to the review as provided in Level 2, except that it will provide a total overview of the data package to ensure its consistency and compliance with project requirements. All errors noted will be corrected and documented.

4.2 Analytical Data Verification and Validation

Sundance will subcontract a third party data validator utilizing established data validation procedures (manually or automated) to perform EPA 100% review and Level III data validation. The review will be performed for drinking water, and soil vapor analytical data obtained from each of the field tasks.

The data review will be performed using the QC criteria specified in the following analytical method and data validation guidelines:

- Project-specific QAPjP
- *DoD Quality Systems Manual for Environmental Laboratories, Version 5.0* (July 2013)
- *USEPA Test Methods for Evaluating Solids Waste, Physical/Chemical Methods* (SW 846, 2006 and updates)
- *USEPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-15, Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* (January 1999)
- MA DEP, *Method for the Determination of Extractable Petroleum Hydrocarbons (EPH)* (May 2004a)
- MA DEP, *Method for the Determination of Volatile Petroleum Hydrocarbons (VPH)* (May 2004b)
- MA DEP, *Method for the Determination of Air-Phase Petroleum Hydrocarbons (APH)* (December 2009)
- American Public Health, Association, American Water Works Association, and Water Environment Federation, *Standard Methods for the Examination of Water and Wastewater, 21st Edition* (2005)
- *USEPA Contract Laboratory Program, National Functional Guidelines for Superfund Organic Methods Data Review* (August 2014)

- *USEPA Contract Laboratory Program, National Functional Guidelines for Inorganic Superfund Data Review, Final* (August 2014)

The following QC elements will be included in the EPA 100% review and Level III data validation:

- Sample extraction and analysis holding times
- Laboratory method blanks
- Surrogate spike recoveries
- LCS/LCSD recoveries
- MS/MSD recoveries
- Laboratory Duplicate, LCS/LCSD and MS/MSD Relative percent differences (RPD)
- Initial calibrations
- Continuing and initial calibration verifications
- Trip, rinse, and ambient field blank results
- Field duplicate sample precision
- For GCMS:
    - Instrument Tune
    - Internal Standards
- Serial Dilutions

Data will be validated and flagged with the following data qualifiers as applicable:

- **J+ qualifier** denotes the analyte was positively identified, but the associated numerical value is estimated with a potential high bias.
- **J- qualifier** denotes the analyte was positively identified, but the associated numerical value is estimated with a potential low bias.
- **U qualifier** denotes the analyte was analyzed for, but was not detected above the MDL.
- **UJ qualifier** denotes that the analyte was not detected above the reported sample LOQ; however, the reported LOQ is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- **R qualifier** denotes the data are unusable due to deficiencies in the ability to analyze the sample and meet QC criteria and DQOs.

As a result of the Level III data validation process, EPA qualifiers will be generated and applied to the affected sample results that exceeded the established QC criteria. EPA 100% review and level III data validation findings will be summarized and documented with each monitoring report.

4.3 Reconciliation with User Requirements

Based on data review and data qualification, the Data Validator will determine if the project DQOs have been met, and data completeness will be calculated. To reconcile the collected data with project DQOs and to establish and document data usability, the data will be reviewed against data quality indicators discussed below.

The Data Validator will prepare a data quality assessment (DQA) report for each of the monitoring events. The DQA report will document:

- Implementation of sampling design and analysis according to the approved QAPjP (or sample completeness and representativeness)
- Proper frequency of field QC samples and the adequacy of field decontamination procedures
- Accuracy and precision of the data
- Data comparability, if applicable
- Data usability for project decisions

4.3.1 Data Quality Indicators

This section defines the data quality indicators and their use for assessment of data quality. These indicators include the PARCC parameters of precision, accuracy, representiveness, comparability and completeness.

4.3.1.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions. The following equation illustrates the method for calculating relative percent difference (RPD) to assess a method's precision:

Precision as RPD	=	$\frac{\text{Absolute (Result – Duplicate Result)}}{\text{Average (Result + Duplicate Result)}}$	x	100%

The laboratory uses MS/MSD samples to assess the precision of analytical procedures. According to USACE requirements, analytical laboratories perform MS/MSD on the project samples to determine whether matrix interferences may be present.

In addition, LCS/LCSD samples can be used to determine analytical method precision when MS/MSD samples are not practical due to the nature of sample or analytical method used. Laboratories will use precision limits specified in the DoD QSM for both LCS and MS analyses (DoD, 2013). When precision limits are not available in the DoD QSM, laboratories may use statistically-based acceptability limits for RPDs established for each method of analysis and sample matrix. The laboratory will review the QC samples to ensure that internal QC data achieve limits of acceptability. Any suspect trends will be investigated and corrective actions taken.

4.3.1.2 Accuracy

Accuracy measures the bias of an analytical system by comparing the difference of a measurement with a reference value. The percent recovery of an analyte, which has been added to the environmental samples at a known concentration before extraction and analysis, provides a quantitation tool for analytical accuracy. The spiking solutions used for accuracy determinations are not used for instrument calibrations. The following equation illustrates how accuracy is evaluated:

Accuracy as Percent Recovery	=	$\frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Spiked Sample True Value}}$	x	100%

Percent recoveries for MS, MSD, and LCS serve as a measure of analytical accuracy. Surrogate standards are added to all samples, blanks, MS, MSD, and LCS analyzed for gas chromatography and mass spectrometry analytical methods to evaluate accuracy of the method and help to determine matrix interferences.

Laboratories will use LCS limits specified in the DoD QSM for both LCS and MS analyses (DoD 2013). When LCS limits are not available in the DoD QSM, the laboratory may use in-house, statistically-based, control limits or control limits specified in EPA methods.

4.3.1.3 Representativeness

Unlike precision and accuracy, which can be expressed in quantitative terms, representativeness is a qualitative parameter. Representativeness is the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. A qualitative parameter depends on proper design of the sampling program.

Field personnel will be responsible for ensuring that samples are representative of field conditions by collecting and handling samples according to the approved QAPjP and WP. Errors in sample collection, packaging, preservation, or COC procedures may result in samples being judged non-representative and may form a basis for rejecting the data.

Data generated by the laboratory must be representative of the laboratory database of accuracy and precision measurements for analytes in different matrices. Laboratory procedures for sample preparation will ensure that aliquots used for analysis are representative of the whole sample. Aliquots to be analyzed for volatile parameters (if any) will be removed before the laboratory composites/homogenizes the samples, to avoid losing volatile compounds during mixing.

4.3.1.4 Comparability

Comparability is a qualitative parameter expressing the confidence where one data set can be compared with another, whether it was generated by a single laboratory or during laboratory studies. The use of standardized field and analytical procedures ensures comparability of analytical data.

Sample collection and handling procedures will adhere to EPA-approved protocols. Laboratory procedures will follow standard analytical protocols, use standard units and standardized report formats, follow the calculations as referenced in approved analytical methods, and use a standard statistical approach for QC measurements.

4.3.1.5 Completeness

Completeness goals for each sampling round are defined in the following section.

4.3.1.5.1 Contractual Completeness

The contractual completeness goal is set at 95% for all methods and is calculated as defined below. The following QC elements are evaluated for the purpose of determining completeness calculation:

- Holding time
- Laboratory blank contamination
- Initial calibration verification
- Continuing calibration verification
- LCSs

% Contract Completeness	=	Number of Unqualified Results* / Number of Results Reported	x	100%

* *Determined by subtracting the results qualified based on contractual deficiencies from the total number of results*

4.3.1.5.2 Analytical Completeness

The analytical completeness goal is set at 90% for all methods and is calculated as defined below. The following QC elements will be considered analytical deficiencies for the purposes of the analytical completeness calculation:

- Holding time
- Laboratory blank contamination
- Field blank contamination (trip, equipment, ambient, and rinse)
- Initial calibration verification
- Continuing calibration verification
- LCS recovery
- MS recovery
- MS precision
- Surrogate recovery

% Analytical Completeness	=	Number of Unqualified Results* / Number of Results Reported	x	100%

* *Determined by subtracting results qualified for any of the deficiencies from the total number of results.*

4.3.1.5.3 Technical Completeness

The technical completeness goal is set at 95% for all methods and is calculated as defined below. Results considered unusable (or rejected) for the intended purpose based on contractual or technical deficiencies will be included for the purposes of the technical completeness calculation:

% Technical Completeness	=	Number of Useable Results* / Number of Results Reported	X	100%

** Technical completeness (i.e., usability) will be determined by subtracting results rejected for any reason from the total number of results reported.*

4.3.2 Project-Required Reporting Limits – Sensitivity

Following the DoD QSM requirements, the laboratory will determine the method detection limits (MDL) for each method, instrument, analyte, and matrix by using the procedure described in Title 40 Code of Federal Regulations Part 136B. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

Following MDL studies, the laboratory will establish the reporting limit or LOQ for each method, analyte, matrix, and instrument in accordance with the DoD QSM requirements. The LOQ is the lowest concentration of a substance that produces a quantitative result within specific limits of precision and bias. The laboratory will perform LOQ verifications to verify precision and bias at the LOQ. The LOQ is greater than the LOD and must be within the calibration range prior to sample analysis. For this project, the laboratory will report positive results down to the MDL and results between the DL and LOQ will be flagged with a J-qualifier and reported as estimated data.

5 ASSESSMENT AND OVERSIGHT

Performing assessments and conducting QA oversight of project activities are vital to verifying that project objectives are being met and assuring the continued quality of the work performed. Assessments will take the form of field surveillances. QA oversight will regularly be performed onsite and is intended to be an interactive part of the field work performed. QA oversight will be performed by the Sundance Project Technical Lead, or designee. QA oversight includes inspections of work performed, verification of field documentation, and site walk-downs.

5.1 Quality Assurance Assessments

Independent assessments shall be planned and conducted to measure item and service quality, evaluate the adequacy of work performance, and promote improvement. The purpose of these assessments is to evaluate the performance of work processes with regard to regulatory, contract, and project requirements and expectations of the client. The group performing independent assessments shall have sufficient authority and freedom from the Sundance project staffing and management line to carry out its responsibilities. Persons conducting independent assessments must be technically qualified and knowledgeable in the areas assessed.

The independent assessment program may include periodic field surveillances of field activities (e.g., soil vapor and drinking water sampling, etcetera). Special emphasis will focus on areas with the highest risk and the greatest benefit from improvement. The surveillance processes will consist of monitoring or observing an item, activity, system, or process to verify that it conforms to specified requirements. These types of assessments are intended to facilitate the frequent monitoring of work in progress to determine and document compliance with established requirements and procedures.

5.2 Quality Assurance Oversight

QA oversight will be performed onsite and is intended to be an interactive part of the field work performed. QA oversight will be performed by the Sundance QA Lead or designee. QA oversight includes inspections of work performed, verification of field documentation, and site walk-downs.

5.2.1 Inspections

Inspection activities will be used to monitor project activities and materials to ensure compliance with established requirements. The objective of inspections is to determine whether the properties, composition, and performance of activities or materials are within established requirements. Inspections shall be performed periodically during the work process to prevent unintended use or installation, to provide monitoring, to minimize delays in work, and to identify nonconformances while they are still correctible without impacting work.

5.2.2 Verification of Field Documentation

Field documentation (e.g., Field Activity Daily Logs, Sample Collection Logs, etcetera) will be reviewed and verified for accuracy and completeness on a regular basis. This verification process is an informal process performed as part of report preparation; allowing for the quick and efficient correction of documentation deficiencies.

5.2.3 Site Walk-downs

Site walk-downs are informal observations of field work being performed. The intent of a site walk-down is to verify that the work is being performed as planned in a safe and orderly manner. Any deficiencies

identified during a walk-down are immediately pointed out to the field crew and corrected. Walk-downs are performed on a daily basis by the Technical Lead/SSHO, but may also be performed by the Sundance PM, or any other senior Sundance personnel.

5.3 Nonconformances and Response Actions

Processes for detecting, preventing, and correcting quality problems are discussed in this section. Items and processes that do not meet established criteria shall be identified, controlled, and corrected, as applicable. Personnel at all levels are responsible for identifying problems and process improvement opportunities and are encouraged to offer solutions.

5.3.1 Problem Identification and Reporting

It is the responsibility of all Sundance and subcontractor personnel to assess activities and inspect items used within the project to verify that each meets specified requirements and to document incidences of nonconforming items, activities, or conditions on a Nonconformance Report (NCR) (Attachment D). It is the responsibility of the project management staff to promptly report, respond to, and resolve nonconforming conditions and to foster a "no-fault" attitude that encourages the identification of nonconforming items and processes.

Personnel who identify a nonconforming condition that is potentially hazardous to workers, the public, or the environment or that jeopardizes the integrity of the program or project have the responsibility and authority to suspend work and report the condition to the responsible manager.

5.3.2 Control and Disposition of Nonconforming Items

Items that do not meet specified requirements, known as nonconforming items, shall be identified by marking, tagging, or other methods that do not adversely affect their end use. Nonconforming items shall be segregated, when practical, by placing them in a clearly identified and designated hold area until properly dispositioned. If segregation is impractical or impossible due to physical conditions, then other administrative controls and precautions should be employed to preclude inadvertent use of nonconforming items.

5.3.3 Nonconforming Activities

Activities or documentation identified as out of compliance with requirements shall be documented as a nonconformance for the purpose of identification of corrective actions and evaluation of the effect on the project objectives. When the integrity of the work is left in question, the work should be performed again, if possible. When not possible, limitations of the results of the work must be documented in the final report of the work.

5.3.4 Cause Analysis

Cause analysis will be performed whenever the understanding of the basic underlying cause is important to the prevention of similar or related problems or when the nonconformance relates to safety. The extent of the cause analysis should be based on the possible negative consequences of a repeat occurrence of a problem. A cause analysis will be used to gain an understanding of the deficiency, its causes, and the necessary corrective actions to prevent recurrence. This analysis should be a systematic process of investigation that uncovers the most basic cause. A summary of the cause analysis shall be documented on the NCR.

5.3.5 Corrective Actions

Responsible managers shall develop and document corrective actions, as applicable, for identified nonconformances. Corrective actions should be targeted at the primary causes of the problem rather than the resulting conditions or secondary causes. These actions shall be reviewed for adequacy and effectiveness in correcting the problem and approved by the PM or a designee.

5.3.6 Improvements and Efficiencies

It is important to identify and report process improvements and efficiency gains. Successful techniques and processes will be evaluated by the Sundance PM, or designee, to determine the potential for performance improvements in other areas or projects.

5.4 Reports to Management

Reports to management may include assessment reports, inspection reports, and NCRs.

5.4.1 Assessment Reports

Surveillance activities will be documented in surveillance reports. Surveillance reports will identify the project activities that were observed/reviewed, the associated requirements documents, and the results of the surveillances, including deficiencies identified and noteworthy practices. Surveillance reports will be prepared/approved by the Sundance Corporate Quality Control Manager (QCM) and presented to the PM within 30 days of performance. Surveillance checklists used during the performance of the surveillance may be included with the final surveillance report. A copy of the final surveillance report shall be placed in the project files.

5.4.2 Nonconformance Reports

Nonconformance reporting will include a description of the nonconforming item or activity, a summary of the corrective action to be taken, assignment of who is responsible for completing the corrective action, and verification that the corrective action is completed. Nonconformance reports will be tracked by the Sundance QA Manager and evaluated by the Sundance PM. A copy of the NCR shall be placed in the project files.

6 DATA MANAGEMENT

Data management is discussed in Section 6.0 of the WP to which this QAPjP is included as an appendix. The WP provides the data management process and procedures to be implemented for the field and for handling laboratory data generated from work activities in support of SVM and drinking water monitoring.

7 REFERENCES

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ATTACHMENT A

LABORATORY ANALYTICAL METHOD REPORTING LIMITS

Table A-1. Method Reporting Limits – Drinking Water

Table A-2. Method Reporting Limits – Soil Vapor

Table A-1. Method Reporting Limits – Drinking Water (TestAmerica, Savannah, GA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Analytical Method	Analyte	CAS Number	Units	NMWQCC [1]	EPA MCL [2]	EPA Tap water		Project Screening Level [4]	Achievable Laboratory Limits [5]		
						RSL [3]	c/nc		LOQ	LOD	MDL
BTEX by EPA 524.2	Benzene	71-43-2	µg/L	10.00	5.00	4.500	c	5.00	0.500	0.250	0.0820
	Ethylbenzene	100-41-4	µg/L	750.00	700.00	15.000	c	700.00	0.500	0.250	0.0990
	m, p-Xylenes	179601-23-1	µg/L	NS	10,000.00	190.000	nc	10,000.00	0.500	0.250	0.0860
	o-Xylene	95-47-6	µg/L	NS	10,000.00	190.000	nc	10,000.00	0.500	0.250	0.0860
	Toluene	108-88-3	µg/L	750.00	1000.00	1100.000	nc	750.00	0.500	0.250	0.0860
EDB by EPA 504.1	Ethylene dibromide	1832-54-8	µg/L	0.10	0.05	0.075	c	0.05	0.0180	0.00500	0.00220

NOTES:
[1] NMWQCC standards per the New Mexico Administrative Code Title 20.6.2.3101A, Standards for Ground Water of 10,000 mg/L Total Dissolved Solids Concentration or Less (NMAC 2004).
For metals, the NMWQCC standard applies to dissolved metals and total mercury.
[2] EPA National Primary Drinking Water Regulations, Maximum Contaminant Levels and Secondary Maximum Contaminant Levels, Title 40CFR Part 141, 143 (May 2009).
[3] EPA Region 6 Regional Screening Levels for Tap water (June 2015) for hazard index = 1.0 for noncarcinogens and a 10^{-5} cancer risk level for carcinogens.
[4] The project screening level was selected to satisfy the requirements of the KAFB Hazardous Waste Permit No. NM9570024423 as the lowest of 1) NMWQCC standard or 2) EPA MCL.
If no MCL or NMWQCC standard exists for any analyte, then the project screening level will be the EPA Tap water RSL.
[5] Achievable laboratory limits are for TestAmerica Laboratories, Savannah, Georgia.

Acronyms and Abbreviations:
µg/L = Microgram(s) per liter
mg/L = Milligram(s) per liter
c/nc = Carcinogenic/ noncarcinogenic
CA = California
CAS = Chemical Abstracts Service

Table A-1. Method Reporting Limits – Drinking Water (TestAmerica, Savannah, GA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 2 of 2)

DL = Detection limit
EDB = ethylene dibromide
EPA = U.S. Environmental Protection Agency
LOD = Limit of detection
LOQ = Limit of quantitation
MCL = Maximum Contaminant Level
MDL = Method Detection Limit
NMWQCC = New Mexico Water Quality Control Commission
NS = no standard
RSL = regional screening level
VOC = volatile organic compound

Table A-2. Method Reporting Limits - Soil Vapor (ALS Global - Environmental, Simi Valley, CA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico

Analytical Group/ Method	Analyte	CAS Number	Units	Project Screening Level	Achievable Laboratory Limits		
					LOQ	LOD	DL
VOCs/TPH EPA TO-15	1,1,1-Trichloroethane	71-55-6	ppbv	Note 1	0.23	0.19	0.078
	1,1,2,2-Tetrachloroethane	79-34-5	ppbv	Note 1	0.18	0.15	0.055
	1,1,2-Trichloroethane	79-00-5	ppbv	Note 1	0.23	0.20	0.073
	1,1-Dichloroethane	75-34-3	ppbv	Note 1	0.31	0.27	0.099
	1,1-Dichloroethene	75-35-4	ppbv	Note 1	0.32	0.28	0.110
	1,2,4-Trichlorobenzene	120-82-1	ppbv	Note 1	0.17	0.15	0.054
	1,2,4-Trimethylbenzene	95-63-6	ppbv	Note 1	0.25	0.22	0.076
	1,2-Dibromoethane	106-93-4	ppbv	Note 1	0.16	0.14	0.052
	1,2-Dichlorobenzene	95-50-1	ppbv	Note 1	0.21	0.18	0.062
	1,2-Dichloroethane	107-06-2	ppbv	Note 1	0.31	0.27	0.099
	1,2-Dichloropropane	78-87-5	ppbv	Note 1	0.27	0.24	0.087
	1,3,5-Trimethylbenzene	108-67-8	ppbv	Note 1	0.25	0.22	0.081
	1,3-Butadiene	106-99-0	ppbv	Note 1	0.57	0.47	0.250
	1,3-Dichlorobenzene	541-73-1	ppbv	Note 1	0.21	0.19	0.062
	1,4-Dichlorobenzene	106-46-7	ppbv	Note 1	0.21	0.17	0.058
	2-Butanone (MEK)	78-93-3	ppbv	Note 1	4.20	0.37	0.180
	2-Hexanone	591-78-6	ppbv	Note 1	0.31	0.27	0.098
	4-Methyl-2-pentanone	108-10-1	ppbv	Note 1	0.31	0.27	0.098
	Acetone	67-64-1	ppbv	Note 1	5.30	2.30	0.810
	Benzene	71-43-2	ppbv	Note 1	0.39	0.35	0.130
	Benzyl Chloride	100-44-7	ppbv	Note 1	0.24	0.22	0.053
	Bromodichloromethane	75-27-4	ppbv	Note 1	0.19	0.16	0.056

Table A-2. Method Reporting Limits- Soil Vapor (ALS Global - Environmental, Simi Valley, CA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 2 of 4)

Analytical Group/ Method	Analyte	CAS Number	Units	Project Screening Level	Achievable Laboratory Limits		
					LOQ	LOD	DL
VOCs/TPH EPA TO-15	Bromoform	75-25-2	ppbv	Note 1	0.12	0.11	0.036
	Bromomethane	74-83-9	ppbv	Note 1	0.32	0.26	0.120
	Carbon Disulfide	75-15-0	ppbv	Note 1	4.00	0.34	0.120
	Carbon Tetrachloride	56-23-5	ppbv	Note 1	0.20	0.18	0.060
	Chlorobenzene	108-90-7	ppbv	Note 1	0.27	0.24	0.087
	Chloroethane	75-00-3	ppbv	Note 1	0.47	0.38	0.160
	Chloroform	67-66-3	ppbv	Note 1	0.26	0.23	0.087
	Chloromethane	74-87-3	ppbv	Note 1	0.61	0.48	0.180
	cis-1,2-Dichloroethene	156-59-2	ppbv	Note 1	0.32	0.28	0.100
	cis-1,3-Dichloropropene	10061-01-5	ppbv	Note 1	0.28	0.23	0.077
	Cyclohexane	110-82-7	ppbv	Note 1	0.73	0.62	0.210
	Dibromochloromethane	124-48-1	ppbv	Note 1	0.15	0.13	0.047
	Dichlorodifluoromethane (CFC 12)	75-71-8	ppbv	Note 1	0.25	0.19	0.086
	Ethyl Acetate	141-78-6	ppbv	Note 1	0.69	0.60	0.240
	Ethylbenzene	100-41-4	ppbv	Note 1	0.29	0.25	0.092
	Hexachlorobutadiene	87-68-3	ppbv	Note 1	0.12	0.11	0.033
	m,p-Xylenes	179601-23-1	ppbv	Note 1	0.58	0.50	0.170
	Methyl tert-Butyl Ether	1634-04-4	ppbv	Note 1	0.35	0.31	0.120
	Methylene Chloride	75-09-2	ppbv	Note 1	0.36	0.32	0.120
	Naphthalene	91-20-3	ppbv	Note 1	0.24	0.21	0.086
	n-Heptane	142-82-5	ppbv	Note 1	0.31	0.27	0.100
	n-Hexane	110-54-3	ppbv	Note 1	0.35	0.31	0.110
	o-Xylene	95-47-6	ppbv	Note 1	0.29	0.24	0.086
	Propene	115-07-1	ppbv	Note 1	0.73	0.58	0.200
	Styrene	100-42-5	ppbv	Note 1	0.29	0.26	0.088
	Tetrachloroethene	127-18-4	ppbv	Note 1	0.18	0.15	0.052
	Tetrahydrofuran (THF)	109-99-9	ppbv	Note 1	0.42	0.37	0.170

Table A-2. Method Reporting Limits- Soil Vapor (ALS Global - Environmental, Simi Valley, CA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Continued, Page 3 of 4)

Analytical Group/ Method	Analyte	CAS Number	Units	Project Screening Level	Achievable Laboratory Limits		
					LOQ	LOD	DL
VOCs/TPH EPA TO-15	Toluene	108-88-3	ppbv	Note 1	0.33	0.29	0.110
	trans-1,2-Dichloroethene	156-60-5	ppbv	Note 1	0.32	0.26	0.120
	trans-1,3-Dichloropropene	10061-02-6	ppbv	Note 1	0.28	0.23	0.088
	Trichloroethene	79-01-6	ppbv	Note 1	0.23	0.20	0.065
	Trichlorofluoromethane	75-69-4	ppbv	Note 1	0.22	0.19	0.076
	Trichlorotrifluoroethane	76-13-1	ppbv	Note 1	0.16	0.14	0.055
	Vinyl Acetate	108-05-4	ppbv	Note 1	3.60	1.50	0.460
	Vinyl Chloride	75-01-4	ppbv	Note 1	0.49	0.39	0.170
APH Method MA DEP	C5-C8 Aliphatic Hydrocarbons	NA	µg/m^3	Note 1	50.00	NA	NA
	C9-C12 Aliphatic Hydrocarbons	NA	µg/m^3	Note 1	25.00	NA	NA
	C9-C10 Aromatic Hydrocarbons	NA	µg/m^3	Note 1	6.30	NA	NA
Fixed Gases ASTM D2504	Oxygen	7782-44-7	%	Note 1	0.10	NA	NA
	Nitrogen	7727-37-9	%	Note 1	0.10	NA	NA
	Carbon Monoxide	630-08-0	%	Note 1	0.10	NA	NA
	Carbon Dioxide	124-38-9	%	Note 1	0.10	NA	NA
	Methane	74-82-8	%	Note 1	0.10	NA	NA
EDB CARB 422	Ethylene Dibromide	1832-54-8	ppbv	Note 1	0.50	NA	0.180

Notes:
Project comparison limits not established.
In accordance with the U.S. Department of Defense Quality Systems Manual requirements, the most current version of the EPA methods will be implemented for each sampling event.

Table A-2. Method Reporting Limits- Soil Vapor (ALS Global - Environmental, Simi Valley, CA)
Bulk Fuels Facility Area
Kirtland Air Force Base, Albuquerque, New Mexico (Concluded, Page 4 of 4)

Acronyms and Abbreviations:
% = percent
APH = air- phase petroleum hydrocarbon
ASTM = ASTM International
CA = California
CAS = Chemical Abstract Service
DL = Detection Limit
MDL = method detection limit
EPA = U.S. Environmental Protection Agency
LOD = limit of detection
LOQ = limit of quantitation
MA DEP = Massachusetts Department of Environmental Protection
NE = not established
ppbv = parts per billion by volume
RL = reporting limit
TPH = total petroleum hydrocarbon
VOC = volatile organic compound

ATTACHMENT B

LABORATORY METHOD CONTROL LIMITS

B-1. Laboratory Control Limits – Drinking Water

B-2. Laboratory Control Limits – Soil Vapor

Table B-1. Method Reporting Limits - Drinking Water

Method	Analyte	CAS Number	RL	MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %
524.2	Benzene	71-43-2	0.500	0.0820	0.250	ug/L	70	130	30	70	130	30
	Ethylbenzene	100-41-4	0.500	0.0990	0.250	ug/L	70	130	30	70	130	30
	Toluene	108-88-3	0.500	0.0860	0.250	ug/L	70	130	30	70	130	30
	Xylenes, Total	1330-20-7	0.500	0.0860	0.250	ug/L	70	130	30	70	130	30
504.1	Ethylene Dibromide	106-93-4	0.0180	0.00220	0.00500	ug/L	70	130	30	70	130	30

Acronyms and Abbreviations:

% = percent
ug/L = microgram(s) per liter
LCS = laboratory control sample
LOD = limit of detection
LOQ = limit of quantitation
MDL = method detection limit
MRL = method reporting limit
MS = matrix spike
NA = not applicable
RPD = relative percent difference

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client:

Client Sample ID: Lab Control Sample ALS Project ID: P1504757

Client Project ID: Kirtland AFB / 140705 ALS Sample ID: P151119-LCS

Test Code:	Massachusetts APH, Revision 1, December 2009	Date Collected:	NA
Instrument ID:	Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13	Date Received:	NA
Analyst:	Evelyn Alvarez	Date Analyzed:	11/19/15
Sample Type:	1.0 L Bottle-Vac™	Volume(s) Analyzed:	0.125 Liter(s)

Test Notes:

Compound	Spike Amount µg/m³	**Result µg/m³**	**% Recovery**	ALS Acceptance Limits	**Data Qualifier**
C5 - C8 Aliphatic Hydrocarbons	216	**200**	**93**	70-130	
C9 - C12 Aliphatic Hydrocarbons	202	**197**	**98**	70-130	
C9 - C10 Aromatic Hydrocarbons	422	**388**	**92**	70-130	

Verified By:_____Date:_____

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client:

Client Sample ID: **Method Blank**
Client Project ID: **Kirtland AFB / 140705**

ALS Project ID: P1504757
ALS Sample ID: P151119-MB

Test Code:	Massachusetts APH, Revision 1, December 2009	Date Collected:	NA
Instrument ID:	Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13	Date Received:	NA
Analyst:	Evelyn Alvarez	Date Analyzed:	11/19/15
Sample Type:	1.0 L Bottle-Vac™	Volume(s) Analyzed:	0.40 Liter(s)

Test Notes:

Compound	**Result µg/m³**	MRL µg/m³	**Data Qualifier**
C_5 - C_8 Aliphatic Hydrocarbons[1,2]	50	50	**U**
C_9 - C_{12} Aliphatic Hydrocarbons[1,3]	25	25	**U**
C_9 - C_{10} Aromatic Hydrocarbons	6.3	6.3	**U**

Significant non-petroleum related peaks (i.e. halogenated, oxygenated, terpenes, etc.) are subtracted from the hydrocarbon range areas when present.

[1]Hydrocarbon Range data from total ion chromatogram excluding any internal/tuning standards eluting in that range.

[2]C_5-C_8 Aliphatic Hydrocarbons exclude the concentration of Target APH analytes eluting in that range.

[3]C_9-C_{12} Aliphatic Hydrocarbons exclude concentration of Target APH Analytes eluting in that range and concentration of C_9-C_{10} Aromatic Hydrocarbons.

ND = Compound was analyzed for, but not detected above the laboratory reporting limit.

MRL = Method Reporting Limit - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client:

Client Project ID: Kirtland AFB / 140705

ALS Project ID: P1504757

1,2-Dibromoethane

Test Code: CARB 422 Modified
Instrument ID: HP5890 II/GC21/ECD
Analyst: Madeleine Dangazyan
Sample Type: 1.0 L Bottle-Vac™(s)
Test Notes:

Date(s) Collected: 11/2/15
Date Received: 11/5/15
Date Analyzed: 11/13/15

Client Sample ID	ALS Sample ID	Injection Volume ml(s)	Canister Dilution Factor	**Result µg/m³**	MRL µg/m³	MDL µg/m³	**Result ppbV**	MRL ppbV	MDL ppbV	Data Qualifier
VA5432	P1504757-001	1.0	1.61	6.2	6.2	2.2	0.81	0.81	0.29	**U**
VA5433	P1504757-002	1.0	1.57	**11**	6.0	2.2	**1.4**	0.79	0.28	
VA5434	P1504757-003	1.0	1.60	6.1	6.1	2.2	0.80	0.80	0.29	**U**
Method Blank	P151113-MB	1.0	1.00	3.8	3.8	1.4	0.50	0.50	0.18	**U**

U = Compound was analyzed for, but not detected above the laboratory reporting limit.
MRL = Method Reporting Limit - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client:

Client Sample ID: **Lab Control Sample**
Client Project ID: **Kirtland AFB / 140705**

ALS Project ID: P1504757
ALS Sample ID: P151113-LCS

Test Code: CARB 422 Modified
Instrument ID: HP5890 II/GC21/ECD
Analyst: Madeleine Dangazyan
Sample Type: 1.0 L Bottle-Vac™
Test Notes:

Date Collected: NA
Date Received: NA
Date Analyzed: 11/13/15
Volume(s) Analyzed: NA ml

CAS #	Compound	Spike Amount ppbV	Result ppbV	% Recovery	ALS Acceptance Limits	Data Qualifier
106-93-4	1,2-Dibromoethane	50.0	**38.2**	**76**	70-130	

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client:

Client Sample ID: **Method Blank**
Client Project ID: **Kirtland AFB / 140705**

ALS Project ID: P1504757
ALS Sample ID: P151120-MB

Test Code: EPA Method 3C Modified
Instrument ID: HP5890 II/GC1/TCD
Analyst: Nalini Lall
Sample Type: 1.0 L Bottle-Vac™
Test Notes:

Date Collected: NA
Date Received: NA
Date Analyzed: 11/20/15
Volume(s) Analyzed: 0.10 ml(s)

CAS #	Compound	Result %, v/v	MRL %, v/v	Data Qualifier
7782-44-7	Oxygen*	0.10	0.10	U
7727-37-9	Nitrogen	0.10	0.10	U
630-08-0	Carbon Monoxide	0.10	0.10	U
74-82-8	Methane	0.10	0.10	U
124-38-9	Carbon Dioxide	0.10	0.10	U

U = Compound was analyzed for, but not detected above the laboratory reporting limit.
MRL = Method Reporting Limit - The minimum quantity of a target analyte that can be confidently determined by the referenced method.
* = The oxygen result may include argon due to coelution. Ambient air includes 0.93% argon.

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client:

Client Sample ID: **Lab Control Sample**
Client Project ID: **Kirtland AFB / 140705**

ALS Project ID: P1504757
ALS Sample ID: P151120-LCS

Test Code: EPA Method 3C Modified
Instrument ID: HP5890 II/GC1/TCD
Analyst: Nalini Lall
Sample Type: 1.0 L Bottle-Vac™
Test Notes:

Date Collected: NA
Date Received: NA
Date Analyzed: 11/20/15
Volume(s) Analyzed: NA ml(s)

CAS #	Compound	Spike Amount ppmV	**Result ppmV**	**% Recovery**	ALS Acceptance Limits	Data Qualifier
7782-44-7	Oxygen*	219,000	**219,000**	**100**	84-121	
7727-37-9	Nitrogen	781,000	**779,000**	**100**	88-122	
630-08-0	Carbon Monoxide	2,000	**2,310**	**116**	87-118	
74-82-8	Methane	1,600	**1,680**	**105**	85-116	
124-38-9	Carbon Dioxide	2,000	**2,150**	**108**	84-117	

* = The oxygen result may include argon due to coelution. Ambient air includes 0.93% argon.

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client:

Client Sample ID: **Lab Control Sample** ALS Project ID: P1504757

Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-LCS

Test Code: EPA TO-15 Modified Date Collected: NA

Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA

Analyst: Evelyn Alvarez Date Analyzed: 11/19/15

Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.125 Liter(s)

Test Notes:

CAS #	Compound	Spike Amount ppbV	Result ppbV	% Recovery	DOD Acceptance Limits	Data Qualifier
115-07-1	Propene	114	**119**	**104**	57-136	
75-71-8	Dichlorodifluoromethane (CFC 12)	38.0	**32.4**	**85**	59-128	
74-87-3	Chloromethane	96.9	**76.8**	**79**	59-132	
75-01-4	Vinyl Chloride	78.3	**63.7**	**81**	64-127	
106-99-0	1,3-Butadiene	93.2	**86.8**	**93**	66-134	
74-83-9	Bromomethane	52.0	**46.2**	**89**	63-134	
75-00-3	Chloroethane	75.8	**66.7**	**88**	63-127	
67-64-1	Acetone	454	**424**	**93**	58-128	
75-69-4	Trichlorofluoromethane	38.5	**30.2**	**78**	62-126	
75-35-4	1,1-Dichloroethene	54.5	**49.3**	**90**	61-133	
75-09-2	Methylene Chloride	63.9	**52.7**	**82**	62-115	
76-13-1	Trichlorotrifluoroethane	28.7	**24.4**	**85**	66-126	
75-15-0	Carbon Disulfide	67.5	**46.6**	**69**	57-134	
156-60-5	trans-1,2-Dichloroethene	53.0	**50.3**	**95**	67-124	
75-34-3	1,1-Dichloroethane	52.4	**47.1**	**90**	68-126	
1634-04-4	Methyl tert-Butyl Ether	59.9	**57.0**	**95**	66-126	
108-05-4	Vinyl Acetate	295	**317**	**107**	56-139	
78-93-3	2-Butanone (MEK)	74.6	**73.2**	**98**	67-130	
156-59-2	cis-1,2-Dichloroethene	55.0	**52.5**	**95**	70-121	

Laboratory Control Sample percent recovery is verified and accepted based on the on-column result.
Reported results are shown in concentration units and as a result of the calculation, may vary slightly.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client: **CB&I Federal Services**
Client Sample ID: **Lab Control Sample** ALS Project ID: P1504757
Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-LCS

Test Code: EPA TO-15 Modified Date Collected: NA
Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA
Analyst: Evelyn Alvarez Date Analyzed: 11/19/15
Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.125 Liter(s)
Test Notes:

CAS #	Compound	Spike Amount ppbV	Result ppbV	% Recovery	DOD Acceptance Limits	Data Qualifier
141-78-6	Ethyl Acetate	119	**130**	**109**	65-128	
110-54-3	n-Hexane	60.2	**58.0**	**96**	63-120	
67-66-3	Chloroform	45.9	**40.6**	**88**	68-123	
109-99-9	Tetrahydrofuran (THF)	74.6	**71.8**	**96**	64-123	
107-06-2	1,2-Dichloroethane	52.9	**50.5**	**95**	65-128	
71-55-6	1,1,1-Trichloroethane	38.5	**34.1**	**89**	68-125	
71-43-2	Benzene	70.8	**66.0**	**93**	69-119	
56-23-5	Carbon Tetrachloride	36.6	**31.1**	**85**	68-132	
110-82-7	Cyclohexane	123	**117**	**95**	70-117	
78-87-5	1,2-Dichloropropane	46.8	**43.5**	**93**	69-123	
75-27-4	Bromodichloromethane	32.6	**30.6**	**94**	72-128	
79-01-6	Trichloroethene	40.2	**34.9**	**87**	71-123	
142-82-5	n-Heptane	52.7	**51.1**	**97**	69-123	
10061-01-5	cis-1,3-Dichloropropene	45.8	**46.5**	**102**	70-128	
108-10-1	4-Methyl-2-pentanone	53.7	**57.6**	**107**	67-130	
10061-02-6	trans-1,3-Dichloropropene	46.3	**48.3**	**104**	75-133	
79-00-5	1,1,2-Trichloroethane	39.6	**37.3**	**94**	73-119	
108-88-3	Toluene	57.9	**51.0**	**88**	66-119	
591-78-6	2-Hexanone	53.7	**69.8**	**130**	62-128	**L**
124-48-1	Dibromochloromethane	25.8	**24.3**	**94**	70-130	

Laboratory Control Sample percent recovery is verified and accepted based on the on-column result.
Reported results are shown in concentration units and as a result of the calculation, may vary slightly.
L = Laboratory control sample recovery outside the specified limits, results may be biased high.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

LABORATORY CONTROL SAMPLE SUMMARY

Client: **CB&I Federal Services**
Client Sample ID: **Lab Control Sample** ALS Project ID: P1504757
Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-LCS

Test Code: EPA TO-15 Modified Date Collected: NA
Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA
Analyst: Evelyn Alvarez Date Analyzed: 11/19/15
Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.125 Liter(s)
Test Notes:

CAS #	Compound	Spike Amount ppbV	Result ppbV	% Recovery	DOD Acceptance Limits	Data Qualifier
106-93-4	1,2-Dibromoethane	28.4	**26.3**	**93**	74-122	
127-18-4	Tetrachloroethene	29.8	**25.3**	**85**	66-124	
108-90-7	Chlorobenzene	47.8	**41.8**	**87**	70-119	
100-41-4	Ethylbenzene	50.2	**46.8**	**93**	70-124	
179601-23-1	m,p-Xylenes	98.6	**95.0**	**96**	61-134	
75-25-2	Bromoform	22.1	**17.4**	**79**	66-139	
100-42-5	Styrene	52.2	**48.9**	**94**	73-127	
95-47-6	o-Xylene	48.4	**46.9**	**97**	67-125	
79-34-5	1,1,2,2-Tetrachloroethane	30.6	**30.3**	**99**	65-127	
108-67-8	1,3,5-Trimethylbenzene	43.5	**42.6**	**98**	67-130	
95-63-6	1,2,4-Trimethylbenzene	44.4	**45.6**	**103**	66-132	
100-44-7	Benzyl Chloride	42.5	**43.8**	**103**	50-147	
541-73-1	1,3-Dichlorobenzene	37.9	**35.9**	**95**	65-130	
106-46-7	1,4-Dichlorobenzene	34.6	**31.5**	**91**	60-131	
95-50-1	1,2-Dichlorobenzene	36.6	**36.3**	**99**	63-129	
120-82-1	1,2,4-Trichlorobenzene	31.0	**24.8**	**80**	55-142	
91-20-3	Naphthalene	41.6	**42.3**	**102**	57-138	
87-68-3	Hexachlorobutadiene	21.6	**16.1**	**75**	56-138	

Laboratory Control Sample percent recovery is verified and accepted based on the on-column result.
Reported results are shown in concentration units and as a result of the calculation, may vary slightly.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client:

Client Sample ID: **Method Blank** ALS Project ID: P1504757

Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-MB

Test Code: EPA TO-15 Modified Date Collected: NA

Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA

Analyst: Evelyn Alvarez Date Analyzed: 11/19/15

Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.40 Liter(s)

Test Notes:

Canister Dilution Factor: 1.00

CAS #	Compound	Result ppbV	LOQ ppbV	LOD ppbV	MDL ppbV	Data Qualifier
115-07-1	Propene	0.73	0.73	0.58	0.20	**U**
75-71-8	Dichlorodifluoromethane (CFC 12)	0.25	0.25	0.19	0.086	**U**
74-87-3	Chloromethane	0.61	0.61	0.48	0.18	**U**
75-01-4	Vinyl Chloride	0.49	0.49	0.39	0.17	**U**
106-99-0	1,3-Butadiene	0.57	0.57	0.47	0.25	**U**
74-83-9	Bromomethane	0.32	0.32	0.26	0.12	**U**
75-00-3	Chloroethane	0.47	0.47	0.38	0.16	**U**
67-64-1	Acetone	5.3	5.3	2.3	0.81	**U**
75-69-4	Trichlorofluoromethane	0.22	0.22	0.19	0.076	**U**
75-35-4	1,1-Dichloroethene	0.32	0.32	0.28	0.11	**U**
75-09-2	Methylene Chloride	0.36	0.36	0.32	0.12	**U**
76-13-1	Trichlorotrifluoroethane	0.16	0.16	0.14	0.055	**U**
75-15-0	Carbon Disulfide	4.0	4.0	0.34	0.12	**U**
156-60-5	trans-1,2-Dichloroethene	0.32	0.32	0.26	0.12	**U**
75-34-3	1,1-Dichloroethane	0.31	0.31	0.27	0.099	**U**
1634-04-4	Methyl tert-Butyl Ether	0.35	0.35	0.31	0.12	**U**
108-05-4	Vinyl Acetate	3.6	3.6	1.5	0.46	**U**
78-93-3	2-Butanone (MEK)	4.2	4.2	0.37	0.18	**U**
156-59-2	cis-1,2-Dichloroethene	0.32	0.32	0.28	0.10	**U**

U = Undetected: The associated data value is the limit of quantitation, adjusted by any dilution factor used in the analysis.

LOQ = Limit of Quantitation - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

Verified By:_____Date:_____

Client: **CB&I Federal Services**
Client Sample ID: **Method Blank** ALS Project ID: P1504757
Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-MB

Test Code: EPA TO-15 Modified Date Collected: NA
Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA
Analyst: Evelyn Alvarez Date Analyzed: 11/19/15
Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.40 Liter(s)
Test Notes:

Canister Dilution Factor: 1.00

CAS #	Compound	Result ppbV	LOQ ppbV	LOD ppbV	MDL ppbV	Data Qualifier
141-78-6	Ethyl Acetate	0.69	0.69	0.60	0.24	**U**
110-54-3	n-Hexane	0.35	0.35	0.31	0.11	**U**
67-66-3	Chloroform	0.26	0.26	0.23	0.087	**U**
109-99-9	Tetrahydrofuran (THF)	0.42	0.42	0.37	0.17	**U**
107-06-2	1,2-Dichloroethane	0.31	0.31	0.27	0.099	**U**
71-55-6	1,1,1-Trichloroethane	0.23	0.23	0.19	0.078	**U**
71-43-2	Benzene	0.39	0.39	0.35	0.13	**U**
56-23-5	Carbon Tetrachloride	0.20	0.20	0.18	0.060	**U**
110-82-7	Cyclohexane	0.73	0.73	0.62	0.21	**U**
78-87-5	1,2-Dichloropropane	0.27	0.27	0.24	0.087	**U**
75-27-4	Bromodichloromethane	0.19	0.19	0.16	0.056	**U**
79-01-6	Trichloroethene	0.23	0.23	0.20	0.065	**U**
142-82-5	n-Heptane	0.31	0.31	0.27	0.10	**U**
10061-01-5	cis-1,3-Dichloropropene	0.28	0.28	0.23	0.077	**U**
108-10-1	4-Methyl-2-pentanone	0.31	0.31	0.27	0.098	**U**
10061-02-6	trans-1,3-Dichloropropene	0.28	0.28	0.23	0.088	**U**
79-00-5	1,1,2-Trichloroethane	0.23	0.23	0.20	0.073	**U**
108-88-3	Toluene	0.33	0.33	0.29	0.11	**U**
591-78-6	2-Hexanone	0.31	0.31	0.27	0.098	**U**
124-48-1	Dibromochloromethane	0.15	0.15	0.13	0.047	**U**

U = Undetected: The associated data value is the limit of quantitation, adjusted by any dilution factor used in the analysis.
LOQ = Limit of Quantitation - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

Verified By:_____Date:_____

ALS ENVIRONMENTAL

RESULTS OF ANALYSIS

Client: **CB&I Federal Services**
Client Sample ID: **Method Blank** ALS Project ID: P1504757
Client Project ID: **Kirtland AFB / 140705** ALS Sample ID: P151119-MB

Test Code: EPA TO-15 Modified Date Collected: NA
Instrument ID: Tekmar AUTOCAN/Agilent 5975Binert/6890N/MS13 Date Received: NA
Analyst: Evelyn Alvarez Date Analyzed: 11/19/15
Sampling Media: 1.0 L Bottle-Vac™ Volume(s) Analyzed: 0.40 Liter(s)
Test Notes:

Canister Dilution Factor: 1.00

CAS #	Compound	Result ppbV	LOQ ppbV	LOD ppbV	MDL ppbV	Data Qualifier
106-93-4	1,2-Dibromoethane	0.16	0.16	0.14	0.052	**U**
127-18-4	Tetrachloroethene	0.18	0.18	0.15	0.052	**U**
108-90-7	Chlorobenzene	0.27	0.27	0.24	0.087	**U**
100-41-4	Ethylbenzene	0.29	0.29	0.25	0.092	**U**
179601-23-1	m,p-Xylenes	0.58	0.58	0.50	0.17	**U**
75-25-2	Bromoform	0.12	0.12	0.11	0.036	**U**
100-42-5	Styrene	0.29	0.29	0.26	0.088	**U**
95-47-6	o-Xylene	0.29	0.29	0.24	0.086	**U**
79-34-5	1,1,2,2-Tetrachloroethane	0.18	0.18	0.15	0.055	**U**
108-67-8	1,3,5-Trimethylbenzene	0.25	0.25	0.22	0.081	**U**
95-63-6	1,2,4-Trimethylbenzene	0.25	0.25	0.22	0.076	**U**
100-44-7	Benzyl Chloride	0.24	0.24	0.22	0.053	**U**
541-73-1	1,3-Dichlorobenzene	0.21	0.21	0.19	0.062	**U**
106-46-7	1,4-Dichlorobenzene	0.21	0.21	0.17	0.058	**U**
95-50-1	1,2-Dichlorobenzene	0.21	0.21	0.18	0.062	**U**
120-82-1	1,2,4-Trichlorobenzene	0.17	0.17	0.15	0.054	**U**
91-20-3	Naphthalene	0.24	0.24	0.21	0.086	**U**
87-68-3	Hexachlorobutadiene	0.12	0.12	0.11	0.033	**U**
1330-20-7	Xylenes, Total	0.58	0.58	0.50	0.17	**U**

U = Undetected: The associated data value is the limit of quantitation, adjusted by any dilution factor used in the analysis.
LOQ = Limit of Quantitation - The minimum quantity of a target analyte that can be confidently determined by the referenced method.

Verified By:_____Date:_____

ATTACHMENT C

ALS ENVIRONMENTAL AND TESTAMERICA SAVANNAH QUALITY ASSURANCE MANUAL AND STANDARD OPERATING PROCEDURES

C-1. TestAmerica Quality Assurance Manual, Savannah, Georgia Laboratory (Drinking Water Analyses)

C-2. TestAmerica Standard Operating Procedures, Savannah, Georgia Laboratory (Drinking Water Analyses)

C-3. ALS Environmental Quality Assurance Manual, Simi Valley, California Laboratory (Soil Vapor Analyses)

C-4. ALS Environmental Standard Operating Procedures, Simi Valley, California Laboratory (Soil Vapor Analyses)

Quality Assurance Manual

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Facility Distribution No. ___1_____ **Distributed To: TALS File System Shares**

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Quality Assurance Manual
Approval Signatures

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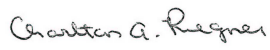

May 12, 2016

Technical Manager, Organics
Charlton Riegner

Date

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CA-Q-S-002	Acceptable Manual Integration Practices
CW-Q-S-003	Internal Auditing
CA-Q-S-006	Detection Limits
CW-Q-S-004	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOP)
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-T-P-001	Qualified Products List
CW-F-S-007	Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health and Safety Manual

LABORATORY SOPs

SOP Reference	Title
SA-AN-041	Reagent and Standard Materials Procedures
SA-AN-100	Laboratory Support Equipment (Verification and Use)
SA-CU-001	Sample Receipt Procedures
SA-CU-015	Preparation of Sampling Kits
SA-EX-015	Toxicity Compound Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP)
SA-EX-030	Liquid Extraction Procedures: Continuous Liquid-Liquid & Separatory Funnel
SA-EX-040	Soil Extraction Procedures: Microwave and Sonication
SA-EX-042	Waste Dilution Extraction
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SA-GE-001	Measurement of Analytes Using Konelab Autoanalyzer
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SA-GE-115	Anions by Ion Chromatography
SA-GE-132	Sulfite
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SA-GE-196	Density and Specific Gravity
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SA-GE-201	Total Hardness as CaCO3 by Titrimetric EDTA
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SA-SM-033	Semivolatile Compounds by GC/MS
SA-VO-001	Preparation, Screening, and Storage of Volatile Samples
SA-VO-002	Volatile Compounds in Drinking Water by GC/MS
SA-VO-003	Acetates in the Pharmaceutical Industry by GC/MS
SA-VO-004	Volatile Compounds by GC/MS
SA-VO-005	Gasoline Range Organics (GRO) by GC/FID
SA-VO-006	Solvents by Direct Aqueous Injection (DAI) Using GC/FID
SA-VO-007	Dissolved Gases in Water

SECTION 3. INTRODUCTION, SCOPE, AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Savannah's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- ANSI/ASQC E4-1994: "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA: 240/B-01/002. May 31, 2006)
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015..*
- *U.S. Department of Defense, Quality Systems Manual (QSM) for Environmental Laboratories, Version 4.2, October 2010.*
- *U.S. Department of Defense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013,*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, and on-line Editions.

- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP),* Version 5

3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control samples. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge, and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in the Methods Listing housed in the laboratory's information management system (i.e., TALS). The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and/or the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the

CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control and Updating procedures (refer to SOP SA-QA-001).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Savannah is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President and Chief Executive Officer (CEO), Chief Operating Officer (COO), Executive Vice President (VP) Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate and TestAmerica Savannah is presented in Figure 4-1.

4.2 Roles and Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Savannah laboratory.

4.2.2 President and Chief Executive Officer (CEO)

The President and CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. The President and CEO establishes the overall quality standard and data integrity program for the Analytical Business, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.3 Chief Operation Officer (COO)

The COO reports directly to the President and CEO of TestAmerica. The COO oversees the operations of all TestAmerica laboratories and the EMLab P&K business unit. The VPs of Operations report directly to COO.

4.2.4 Vice President of Operations

Each VP of Operations reports directly to the Executive VP of Operations and is a part of the Executive Committee. Each VP of Operations is responsible for the overall administrative and operational management of their respective laboratories. The VP's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the

financial, business, and quality objectives of TestAmerica. The VPs ensure timely compliance with Corporate Management directives, policies, and management systems reviews. The VPs are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.5 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to the President and CEO. With the aid of the Executive Committee, Laboratory Directors, Quality Directors, Safety Manager, EH&S Coordinators and QA Managers, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and EH&S Programs within TestAmerica. Additional responsibilities include:

- Review of QA/QC and EHS aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the analytical laboratories and a summary of any quality related initiatives and issues.
- Preparation of a monthly report that includes EH&S metrics across the analytical laboratories and a summary of any EH&S related initiatives and issues.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

4.2.6 Vice President of Client Service

The VP of Client Services leads the Client Service Organization (CSO) and is responsible for client satisfaction, driving operational excellence and improving client responsiveness. The VP provides direction to the Client Service Directors, Programs Managers and Project Managers.

4.2.7 Quality Assessment Director

The Quality Assessment Director reports to the VP-QA/EHS. The Quality Assessment Director has QA oversight of laboratories; is responsible for the internal audit system, schedule and procedure; monitors laboratory internal audit findings; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Compliance Director, the Quality Systems Director, and the VP-QA/EHS, the Quality Assessment Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.8 Quality Compliance Director

The Quality Compliance Director reports to the VP-QA/EHS. The Quality Compliance Director has QA oversight of laboratories; monitors and communicates DoD / DoE requirements; develops corporate tools for ensuring and improving compliance; develops corporate assessment tools; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Systems Director and the VP-

QA/EHS, the Quality Compliance Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.9 Quality Systems Director

The Quality Systems Director reports to the VP-QA/EHS. The Quality Systems Director has QA oversight of laboratories; develops quality policies, procedures and management tools; monitors and communicates regulatory and certification requirements; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Compliance Director and the VP-QA/EHS, the Quality Systems Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.10 Quality Information Manager

The Quality Information Manager is responsible for managing all company official documents (e.g., Policies, Procedures, Work Instructions), the company's accreditation database, intranet websites, external laboratory subcontracting, regulatory limits for clients on the company's TotalAccess website; internal and external client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions. The Quality Information Manager reports to the VP-QA/EHS; and works alongside the Quality Assessment, Quality Compliance and Quality System Directors and EHS Managers to support both the Analytical Quality Assurance and EHS Programs within TestAmerica.

4.2.11 Technical Services Director

The Technical Services Director is responsible for establishing, implementing and communicating TestAmerica's Analytical Business's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

4.2.12 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – Corporate Counsel & VP of Human Resources and the VP-QA/EHS. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President and CEO, VPOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and

processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.13 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

4.2.14 Environmental Health and Safety Managers (Corporate)

The EHS Managers report directly to the VP-QA/EHS. The EHS Managers are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.15 Laboratory Director

The Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective VPO. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program. Specific responsibilities include, but are not limited to, the following:

- Provides one or more Technical Managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.

- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Manager(s), and the Operations Manager as direct reports.

4.2.16 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation and improvement of the quality system based on ISO/IEC 17025, DOD ELAP, and TNI. The QA Manager is independent of production; reports directly to the Laboratory Director and their Corporate Quality Director; and has access to Corporate QA for advice and resources. The QA Manager is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. The QA Manager directs the activities of the QA Department to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities. Ensuring all personnel understand their contributions to the Quality System.
- Having documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintaining records of all ethics-related training, including the type and proof of attendance.

- Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating the document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Reviewing a percentage of all final data reports for consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, format, holding time, sensibility, and completeness of the project file contents.
- Reviewing of external audit reports and data validation requests.
- Following-up with audits to ensure client QAPP requirements are met.
- Establishing of reporting schedule and preparation of various quality reports for the Laboratory Director, clients, and/or Corporate QA.
- Developing of suggestions and recommendations to improve quality systems.
- Researching current state and federal requirements and guidelines.
- Managing the QA team to enable communication and to distribute duties and responsibilities.
- Evaluating of the thoroughness and effectiveness of training.
- Ensuring compliance with ISO/IEC 17025, DOD ELAP, and TNI.

4.2.17 Technical Manager / Director

The Technical Manager(s) report(s) directly to the Laboratory Director. The Technical Manager is accountable for all analyses and analysts under their experienced supervision and for compliance with ISO 17025, DOD ELAP, and TNI. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods (i.e., SOPs) with regard to quality, integrity, regulatory requirements, and optimum and efficient production techniques; and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He ensures that the SOPs are properly managed and adhered to at the bench.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, and the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any

deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.

- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting project QAPPs, ensuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting, and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved TALS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," ensuring that no time is lost in locating samples.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Ensuring compliance with ISO/IEC 17025, DOD ELAP, and TNI.

4.2.18 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He reports directly to the Laboratory Director. He assists the Technical Manager in determining the most efficient instrument utilization**.** Specific responsibilities include, but are not limited to, the following:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Manager and QA Manager and in compliance with regulatory requirements.
- Works with the Preventive Maintenance Coordinator to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system.

4.2.19 Compliance Officer / Environmental Health and Safety Coordinator

The Environmental Health and Safety Coordinator reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. Specific responsibilities include, but are not limited to, the following:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.

- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

4.2.20 Department Manager ~~Supervisor~~

Supervisors report to the Operations Manager. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 8.1), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, investigation of non-conformance issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.

- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.21 Analyst

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.22 Manager of Project Managers (MPM)

Specific responsibilities include, but are not limited to, the following:

- Coordinates marketing efforts with General Manager, Laboratory Director, Project Managers, and laboratory marketing group
- Supervises Project Managers
- Coordinates proposal and contract review and response process
- Responds to client inquiries

4.2.1.10 Project Manager (PM)

The PM reports to the Manager of Project Management (MPM) and serves as the interface between the laboratory's technical departments and the laboratory's clients. There is an entire staff of Project Managers that makes up the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.

- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

4.2.1.11 Custody Supervisor

The Custody Supervisor Manager reports to the Laboratory Director. He is responsible for ensuring the timely and correct shipment of sample containers, including proper preservatives and instructions, to clients. He maintains accurate records of sample container shipments. In addition, he:

- Schedules bottle orders and supervises bottle prep staff
- Supervises sample custody staff
- Coordinates with Project Managers and Field/Sampling Supervisor on scheduling field sampling efforts
- Identifies and documents custody discrepancies and notifies Project Managers about custody problems

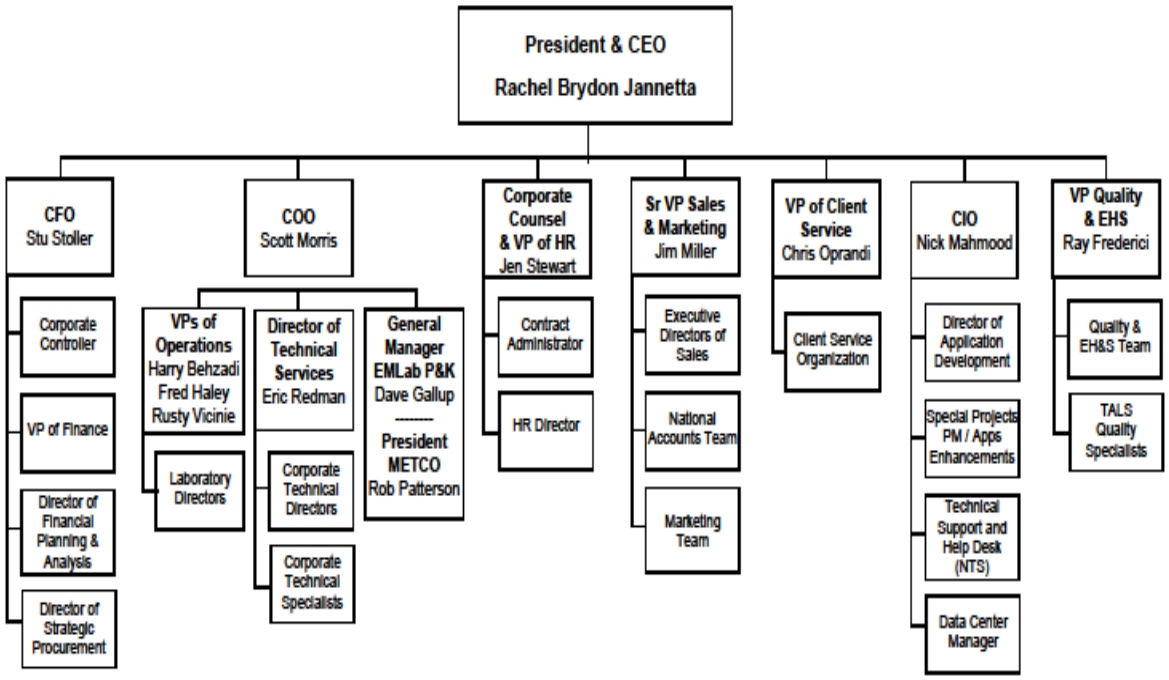
4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Laboratory Director	QA Manager
QA Manager	Laboratory Director
Operations Manager	Laboratory Director
Technical Director/Manager	Laboratory Director
EHS Coordinator	QA Manager

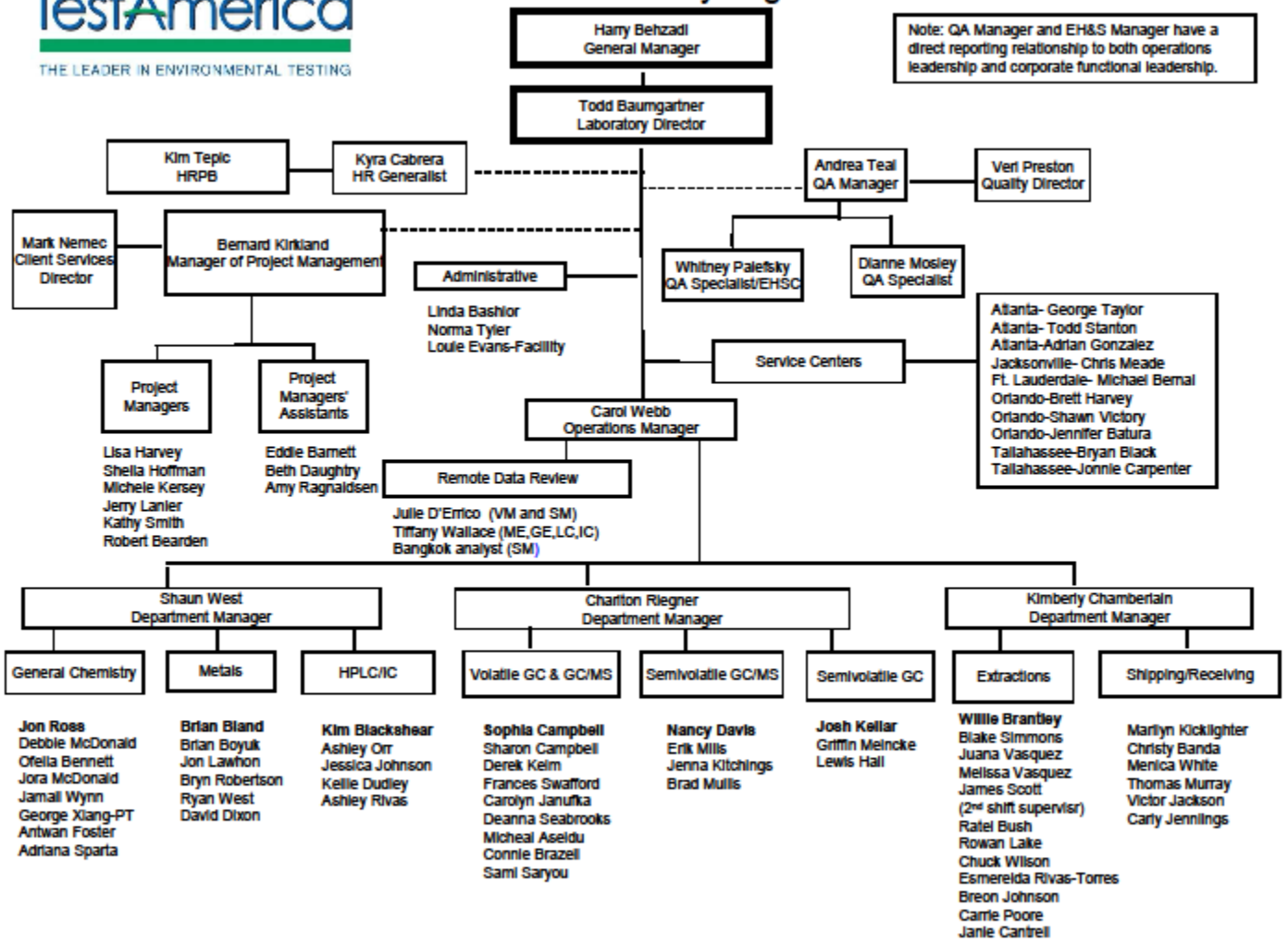
Figure 4-1. Corporate and Laboratory Organization Charts

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Savannah Laboratory Organization



SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- Comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard, and the DOD QSM, and to continually improve the effectiveness of the quality management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics and Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002)
- Procedures and guidance for recalling data if necessary. (Corporate SOP No. CA-Q-S-005)
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).

- Production of results, which are accurate and include QA/QC information that meet client pre-defined Data Quality Objectives (DQOs).
- Presentation of services in a confidential, honest, and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all laboratories. The policies described therein are typically incorporated into laboratory-specific SOPs, or the Corporate documents may be are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPPs) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or matrix spikes (MS). A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and

field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc.

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (e.g., Method Detection Limit) or quantified (e.g., Reporting Limit).

5.5 Criteria for Quality Indicators

The laboratory maintains Method Limit Groups in TALS that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA Department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP SA-QA-017: *Evaluation of Batch QC Data*.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory that are entered into the Laboratory Information Management System (i.e. TALS). The Quality Assurance Department maintains an archive of all limits used within the laboratory and stores these values in TALS. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the laboratory develops such limits from recent data in the QC database of TALS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the TALS analyte database. As sample results and the related QC are entered into TALS, the sample QC values are compared with the limits in TALS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (recommended annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.* The QA Manager evaluates control charts to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

5.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOPs)
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers, and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, *Corporate Document Control and Archiving*. The laboratory's internal document control procedure is defined in SOP SA-QA-001: *Document Control Program*.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, an employee submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System policies and procedures will be reviewed at a minimum of every year and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP refer to SOP SA-QA-001: *Document Control Program.*

Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA Department. Electronic controlled copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, refer to SOP SA-QA-001: *Document Control Program.*

Electronic copies of current documents (including QA Manuals, SOPs, Forms, Work Instructions, etc.) are maintained by the QA Department and distributed electronically via the TALS File System Shares.

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, for hardcopy distribution, obsolete documents are collected from employees according to distribution lists and are destroyed. At least one copy of the obsolete document is archived according to SOP SA-QA-001: *Document Control Program.*

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the laboratory's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals, and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel, and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the laboratory to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements, and that the lab has the capacity to meet the clients turn around needs.

For new, complex or large projects, the proposed contract is given to the Sale Directors, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002: *Contract Compliance Policy.*

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above.

The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the designated personnel is not available to review the contract, his back-up will fulfill the review requirements.

The Contracts Department maintains copies of all signed contracts.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are maintained by the Proposal Coordinator.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA Department involvement may be needed to assist in the evaluation of custom QC requirements.

PMs are the primary client contact and they ensure resources are available to meet project requirements. Although PMs do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each Project in TALS as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, email, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the supervisor.

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO/IEC 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers, Operations Managers, Department Managers, and the QA Manager are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develop laboratory and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOPs on subcontracting procedures (i.e., CA-L-S-002) and the worksharing process (i.e., CA-C-S-001).

When outsourcing analytical services, the laboratory will ensure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI and ISO/IEC 17025, and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs), Client Service Managers (CSM), or Account Executives (AE) for the export lab (i.e., the TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing, and, when possible, approval from the client shall be retained in the project folder. Standard TestAmerica Terms & Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., certain USACE projects) may require notification prior to placing such work.

8.2 Qualifying and Monitoring Subcontractors

Whenever a PM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task. Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an email from the client in the project folder.

- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract laboratory. Verify necessary accreditation, where applicable, (e.g., on the subcontractor's TNI, A2LA accreditation, or State Certification).
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned, and/or minority-owned businesses;
- TNI or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for worksharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an email is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs as specified in Corporate SOP (CA-C-S-001: *Worksharing Process*).

When the potential subcontract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager or PM begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002: *Subcontracting Procedures*.

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. Once all documents are reviewed for completeness, the Corporate QIM will forward the documents to the Purchasing Manager for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Corporate Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the

subcontracted laboratories.

- Subcontractors in good standing will be retained on the intranet listing. CSO personnel will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all CSO personnel, Laboratory Directors, QA Managers, and Sales personnel.

8.3 Oversight and Reporting

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and Corporate Counsel can tailor the document or assist with negotiations, if needed. The PM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it is current and scope-inclusive. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for all samples workshared within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor laboratory. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which laboratory produced the data for which methods and samples.

Note: The results submitted by a TestAmerica worksharing laboratory may be transferred electronically and the results reported by the TestAmerica worksharing laboratory are identified on the final report. The report must explicitly indicate which lab produced the data for which

methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFPs) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFPs allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards, and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent and Acid Lot Testing and Approval, SOP No. CA-Q-S-001. Approval information for the solvents and acids tested under CA-Q-S-001 is stored on the TestAmerica SharePoint under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

9.3.2 Receiving

It is the responsibility of the Shipping and Receiving Department to receive the shipment. Once the materials are received, the laboratory compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date to the information present on the order log.

Materials may not be released for use in the laboratory until they have been inspected and verified as suitable for use. The laboratory verifies the lot numbers of received solvents and acids against the pre-approved lists. If a material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained in TALS.

Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration date noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer or SOP expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date cannot be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of

gas should be replaced when it drops to approximately 500psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and Technical Manager must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots may be verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained electronically.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed, and Purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and it is added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate for the specific intended application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs) if a new method, and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department. Software certificates supplied by the vendors are filed with the TALS Administrator. The manufacturer's operation manual is retained electronically.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP SA-AN-100: *Laboratory Support Equipment*. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed and filed. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers and weight sets are obtained from vendors with current and valid ISO/IES 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department and filed. The equipment is then returned to service within the laboratory.

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal/bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts, or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc. As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors.

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a JD Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or Technical Management are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling process to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations, and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing, and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints, or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SA-QA-005: *Preventive and Corrective Action.*

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP SA-QA-005.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing, and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the laboratory and Quality Director in the QA Monthly Report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies, and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the Department Manager for resolution. The Department Manager may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client requests that a special procedure be applied to a sample that is not standard laboratory practice. Based on a technical evaluation, the laboratory may accept or reject the request based on technical or ethical merit. Such a request would need to be approved by laboratory management and documented in the project files. Deviations to standard operating procedures must be noted in the final report.

11.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to a member of Senior Management within 24 hours. The Senior Management staff is comprised of the Laboratory Director, Operations Manager, QA Manager, and the Technical Manager. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of Operations, and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

Corporate SOP entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-Q-S-005.

11.4 Prevention of NonConforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required, and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may

not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target, or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be provided by the laboratory to the appropriate member of Corporate QA, which serves as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing, or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will not generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, or determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval may be given by final signature on the completed corrective action report.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using NonConformance Memos (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends

- Excessive revised reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a event requiring action, the event must be defined and documented. A CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with the problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager, Operations Manager, and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each CAR is entered into a database for tracking purposes.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis. Refer to Figure 12-1.
- The QA Manager reviews monthly NCRs and CARs for trends. Highlights are included in the QA Monthly Report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, SOP SA-QA-017: *Evaluation of Batch QC Data* and the analytical SOPs.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA Monthly Report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1.
Corrective Action Report

Section 1 **Summary of Problem / Finding**
Finding #: Summary: Date Due to Agency:
Section 2 **Initial Investigation Summary**
Investigation Question #1: • Is this issue chronic (i.e., were multiple instances cited, or is the potential for similar issues present), or acute (i.e., an isolated, anomalous, or non-routine occurrence)? Response:
Investigation Question #2: • Are other departments likely to be impacted? Response:
Investigation Question #3: • Can the root cause be readily established/addressed and action items identified without further inquiry, or is further action needed to perform a formal RCA Investigation and/or develop the Corrective Action Plan? Note: If the root cause can be readily established/addressed and action items identified without further inquiry, then Section 3 does not need to be completed provided additional details are included in response to Investigation Question #4, below. Response:
Investigation Question #4: • Are there any additional comments worth noting? If so, please include. Response:
Section 3 **Root Cause Analysis Summary**
RCA Investigation Lead: RCA Investigation Team Members, if applicable:

RCA Question #1:

- Why was this finding cited?

Options:
1. Procedure/policy does not exist, is not adequate, or is not accurate.
2. Procedure/policy is in place, adequate, and accurate; however, employee did not comply.
3. Other

Response:

RCA Question #2:

- What are some underlying causes for the conclusion drawn in RCA Question #1 (i.e., what are some Quality System weaknesses indicated by this issue that also need to be addressed)?

Note: There may be more than one underlying cause/weakness, and each underlying cause/weakness may in turn have other underlying causes/weaknesses.

Examples:
1. Insufficient or incomplete method validation procedures.
2. Trend analysis was not performed or is insufficient.
3. Insufficient or incorrect detail in SOPs; SOPs out of date; SOPs do not match current practice, etc.
4. Missing or inadequate mechanism to capture information (e.g., form, spreadsheet, Data Types, etc.).
5. Missing or inadequate training.
6. Insufficient employee oversight / supervision.
7. Ineffective primary data review process.
8. Ineffective self-monitoring process (e.g., notebook review, secondary data review, internal audits, etc.).
9. Personnel problem, insufficient resources, lack of attention to detail, etc.
10. Insufficient reagent traceability or control procedures.
11. Poor communication channels.
12. Improper or inadequate equipment maintenance procedures.
13. Ineffective Document Control mechanisms
14. Ineffective sample scheduling mechanisms, workflow, backlogs, etc.
15. Other

Response:

RCA Question #3:

- Is a Data Recall, an SOP revision, or additional training needed?

Response:

RCA Question #4:

- Are there any additional comments worth noting? If so, please include.

Response:

Section 4
Corrective Action Assignments

<<Based on the Initial Investigation and/or Root Cause Analysis Summary outlined above, what action items are needed to: 1) correct the original finding, and 2) minimize its recurrence? >>

Action Item #1:

Assigned Party:
Due Date:
Status:

Actions Taken:

Supporting Documentation Attached:

Section 5
Audit Response Documentation

Laboratory Response sent to agency on: XXXXX, attached here.

Section 6
Subsequent Information / Documentation Requests from Agency

Summary:

Assigned to:
Due Date:

Documentation attached here:

Section 7
Additional Close-Out / Follow-Up and Comments

A)
This finding pertains to an isolated and/or anomalous event. The corrective action taken is sufficient to address this issue. No further action or follow-up is needed at this time to close out this item.
Initial / Date:

B)
An additional routine follow-up assessment is required to evaluate the effectiveness of the corrective action taken.
Follow-up Assigned To:
Due Date:
Documentation Needed:
Items used to assess effectiveness/sustainability of corrective action:
<<Include AD batch numbers, attach example logbook pages, etc., as applicable.>>

Choose One:
a) Corrective action has been implemented and is effective.
b) Similar problems have been noted. The corrective action has not been effective. Additional action is required.
Initial / Date:

Table 12-1. General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Instrument Blank *(Analyst)*	- Criteria in analytical SOP	- Prepare and analyze another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Analyst)*	- Criteria in analytical SOP	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Initial Calibration Verification (Second Source ICV) *(Analyst)*	- Criteria within analytical SOP	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Verification (CCV) *(Analyst)*	- Criteria within analytical SOP	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst)*	- Criteria in TALS MLGs	- If matrix interferences are present, evaluate the LCS. - If the LCS is within acceptable limits the batch is acceptable.
Laboratory Control Sample (LCS) *(Analyst)*	- Criteria in TALS MLGs and SOP SA-QA-017	- Reanalyze LCS. - Batch must be re-prepared and/or re-analyzed.
Surrogates *(Analyst)*	- Criteria in TALS MLGs	- Individual sample must be repeated, unless obvious matrix interference is noted.
Method Blank *(Analyst)*	<1/2RL	- Reanalyze blank. - Determine source of contamination. - Re-prepare/re-analyze batch.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems, and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report
- trending NCMs
- review of control charts and QC results
- trending proficiency testing (PT) results
- performance of management system reviews
- trending client complaints
- review of processing operations
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly be the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lessons Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.

- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions/Process Improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance, and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA Department on the Q-Drive and are backed up as part of the regular network backup. Technical records are maintained by the laboratory departments in the Data Archival folder on the Public_QA Drive and are backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer- or hand-generated (some records may be in both formats).

Table 14-1. Records Index[1]

	Record Types [1]:	Retention Time:
Technical Records	Raw Data Logbooks[2] Standards Certificates Analytical Records MDLs/IDLs/DOCs Lab Reports	5 Years from analytical report issue*
Official Documents	Quality Assurance Manual (QAM) Work Instructions Policies SOPs	5 Years from document retirement date*
QA Records	Internal & External Audits/Responses Certifications Corrective/Preventive Actions Management Reviews Method & Software Validation Data Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	Sample Receipt & COC Documentation Contracts and Amendments Correspondence QAPP SAP Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual and Permits	5 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies Technical Training Records	Refer to HR Manual

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance Log, Instrument Run Log, Preparation Logs (standard and samples), Standard and Reagent Receipt Logs, Balance Calibrations, Temperature Logs, etc.
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2. Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	5 years (project records) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio VAP	10 years and State contacted prior to disposal

Program	[1]Retention Requirement
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 for more information. Electronic records are maintained in the Data Archival Folder on the Public_QA drive, or in another applicable drive (such as Q-drive). Refer to SOP SA-QA-001: *Document Control Program* for specific information on the archival, storage, and back-up of records.

14.1.4 The recordkeeping system allows for historical reconstruction of all laboratory activities that produced the analytical data as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The chain of custody would indicate the name of the sampler.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The recordkeeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set, etc. as per SOP SA-QA-001: *Document Control Program).* Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Where an analysis is performed without an instrument, TALS sheets, bound logbooks, bench sheets, or spreadsheets are used to record and file data. Standard and reagent information is recorded in TALS for each method.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning

process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

14.2 Technical and Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records, and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code**;**
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the TALS and on specific analytical report formats.

14.2.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a documented supervisory or peer review on a monthly basis.

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates, and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the TALS. Records are considered archived when noted as such in the records management system.

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation, or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and**,** when requested**,** to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits / QA Technical Audits	Joint Responsibility: a) QA Manager or designee, b) Technical Manager or designee (Refer to SOP No. CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint Responsibility: a) QA Manager or designee, b) Technical Manager or designee (Refer to SOP No. CW-Q-S-003)	SOP Compliance Review Frequency: - Every 2 years (non-DOD SOPs) - 100% of SOPs annually (DOD SOPs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness and sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., CHROM AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period. All analysts should be reviewed over the course of a two year period through at least one QA Technical Audit.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every year. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities (new IDOC), reviews of the analyst work products will be performed.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

Single blind performance audits are employed for several reasons. One purpose is to provide corrective action for parameters judged to be unacceptable on external or internal performance audits. Periodic internal performance audits are also used to test parameters that are not routinely tested by external performance audits. Finally, single blind performance audits are employed to satisfy certain certification requirements, to satisfy auditors' specific requests for performance audit samples, or to provide additional evidence of data quality to clients with specific questions regarding laboratory performance.

The laboratory participates semi-annually in performance audits conducted through the analysis of Proficiency Testing (PT) samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-Potable Water, and Soil.

These PT studies are performed approximately six months apart. The first study of the year is usually performed in January, and the second study is usually performed in July. The PT results are submitted to certification agencies directly from the PT Provider. Remedial PT studies can be performed, as required, for any analytes scored as unacceptable. Root cause investigation into any unacceptable results must be initiated. Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the laboratory's corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible

laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department and/or Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the laboratory's corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Quality Director, and the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and VPs of Operations.

16.2 Annual Management Review

The senior laboratory management team (Laboratory Director, Operations Manager, QA Manager) conducts a review annually of its quality systems and TALS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives, and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel can be included in this meeting at the discretion of the Laboratory Director. The TALS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the TALS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
- Adequacy of staff, equipment and facility resources.

- Adequacy of policies and procedures.
- Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate VP of Operations and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table).

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Internal Investigations SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's President and CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations, and Quality Directors receive a monthly report from the VP-QA/EHS summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific laboratories.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures, and records management.

Laboratory management is responsible for formulating goals for laboratory staff with respect to education, training, and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the laboratory staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform, or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources webpage. (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Director – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

Specialty	Education	Experience
Technical Director - Microbiology	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics (New Hires)	1 week of hire	All
Ethics (Comprehensive)	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics (Comprehensive Refresher)	Annually	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood, and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques, or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst's knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicating to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the SOP SA-QA-006: *Training Procedures*.

17.4 Data Integrity and Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive ethics training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 55,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control, and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and TALS are regulated to protect against raw data loss.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas available to ensure unencumbered work. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys and alarm codes are distributed to employees as necessary.

Employees wear photographic identification name cards while on the premises.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage, and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOP)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints, as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP SA-QA-001: *Document Control*.
- SOPs are reviewed at a minimum of annually, and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*

- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008, Final Update V, August 2015.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method, or personnel (e.g., analyst has not performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve, and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented, and the laboratory informs the client of its procedure for working with unusual compounds.

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Refer to SOP SA-QA-006: *Training Procedures* for information on performing Initial Demonstrations of Capability (IDOC).

A certification statement (refer to Figure 19-1) can be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper

quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of method performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks, or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value can be differentiated from blanks. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)* for details on the laboratory's MDL process.

19.8 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of instrument blanks and calculating three times the absolute value of the standard deviation.

If IDL is greater than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP SA-QA-008 for further details.

The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.11 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, and chromatography retention time windows.

19.12 Estimation of Uncertainty of Measurement

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty" (i.e., the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2).

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5mg/L.

Refer to SOP SA-QA-017: *Evaluation of Batch QC Data* for more information on this topic.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., EPA 524.2, EPA 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also

variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client-specific, contractual Terms and Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the laboratory was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to non-homogenous samples, Encores/Terracores, and sodium bisulfate preserved samples.

19.14 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the TestAmerica LIMS System (TALS) which is a custom in-house developed TALS system that has been highly customized to meet the needs of the laboratory. It is referred to as TALS for the remainder of this section. TALS utilizes Microsoft SQL Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 Maintain the Database Integrity: Assurance that data are reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal TALS permissions procedure.

- TALS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails, and controlled access.

19.14.1.2 **Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 **Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to approving the data in TALS.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and SOP SA-QA-008: *Evaluation of Chromatographic Data.*

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data is retained in the laboratory benchsheets, computer file (if appropriate), and/or runlog. All criteria pertinent to the method are recorded. The documentation is recorded at the time observations or calculations are made and each person involved is readily identified.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter (ug/L) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (ug/kg) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%. Units are defined in each laboratory SOP.

19.14.2.3 In general, results are reported to 2 significant figures on the final report.

19.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the TALS, the raw results and dilution factors are entered directly into TALS by the analyst, and the software calculates the final result for the analytical report.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with

the TALS, the raw results and dilution factors are transferred into TALS electronically. Electronic data from instruments are saved electronically in a daily folder on the system (CHROM or instrument computer). For instruments that print out calibrations and concentrations, the data are retained with the data file. The data file is stored in the Archival Folder on the Public_QA. These files are transferred to the server daily, and eventually, to a tape file.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Department controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Data review procedures are outlined in the analytical SOPs and SOP SA-QA-002: *Data Generation and Review* and ensure that data reported are free from calculation and transcription errors and that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing manual integrations to ensure the authenticity of the data (SOP SA-QA-008). The general review concepts are discussed below; more specific information can be found in the SOPs.

19.14.4.1 Log-in Review - The data review process at starts at the sample receipt stage. Sample Control personnel review chain-of-custody forms and input the sample information into the TALS. The Project Management Assistant reviews the transaction of the chain-of-custody forms and inputs the required analyses. The Project Managers perform final review of the chain-of-custody forms and entered information.

19.14.4.2 First Level Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analysts transfer the data into TALS and data qualifiers are added as needed. All first level reviews are documented.

19.14.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data

(e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples have unusually high results
- Samples exceed a known regulatory limit
- Raw data indicates some type of contamination or poor technique
- Inconsistent peak integration is observed
- Transcription errors are identified
- Results are outside of calibration range

19.14.4.4 Unacceptable analytical results may require reanalysis of the samples. Problems may be brought to the attention of the Laboratory Director, Project Manager, Operations Manager, Quality Assurance Director/Manager, Technical Manager, or Department Manager for further investigation, if needed. Corrective action is initiated whenever necessary.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/narratives are present, data qualifiers are appropriate, and project-specific requirements are met. The following are some examples of chemical relationships that can be reviewed (if data is available):

- Total Results are $\geq$ Dissolved results (e.g. metals)
- Total Solids (TS) $\geq$ Total Dissolved Solids (TDS) or Total Suspended Solids (TSS)
- TKN $\geq$ Ammonia
- Total Phosphorus $\geq$ Orthophosphate
- COD $\geq$ TOC
- Total Cyanide $\geq$ Amenable Cyanide
- TDS $\geq$ individual anions

19.14.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report and sends to the client.

19.14.4.7 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal SOP No. SA-QA-008, entitled *Evaluation of Chromatographic Data*.

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integration is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Demonstration of Capability Documentation

TRAINING DOCUMENTATION FORM
DEMONSTRATION OF CAPABILITY

Laboratory Name: TestAmerica Savannah
Address: 5102 LaRoche Avenue
Savannah, GA 31404

Date Completed: _____

Analyst Name: _____

Prep Analyst Name (s): _____

Analytical Test Method: _____

Prep Method: _____

Matrix: ☐ Soil ☐ Aqueous ☐ Other

Analytical SOP Document Control Number: _____

Prep SOP Document Control Number: _____

Analyte, Class of Analytes, or Measured Parameters: _____

If PT Study is used as DOC, list the PT Number: _____

We, the undersigned, CERTIFY that:
1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program and/or other state and federal programs have completed the Demonstration of Capability.
2. The test method(s) was performed by the analyst(s) identified on this certification.
3. A copy of test method(s) and laboratory-specific SOPs are available for all personnel on-site.
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

Technical Director's Name	Signature	Date
Quality Assurance Officer's Name	Signature	Date

FQA049:08.13.07:6

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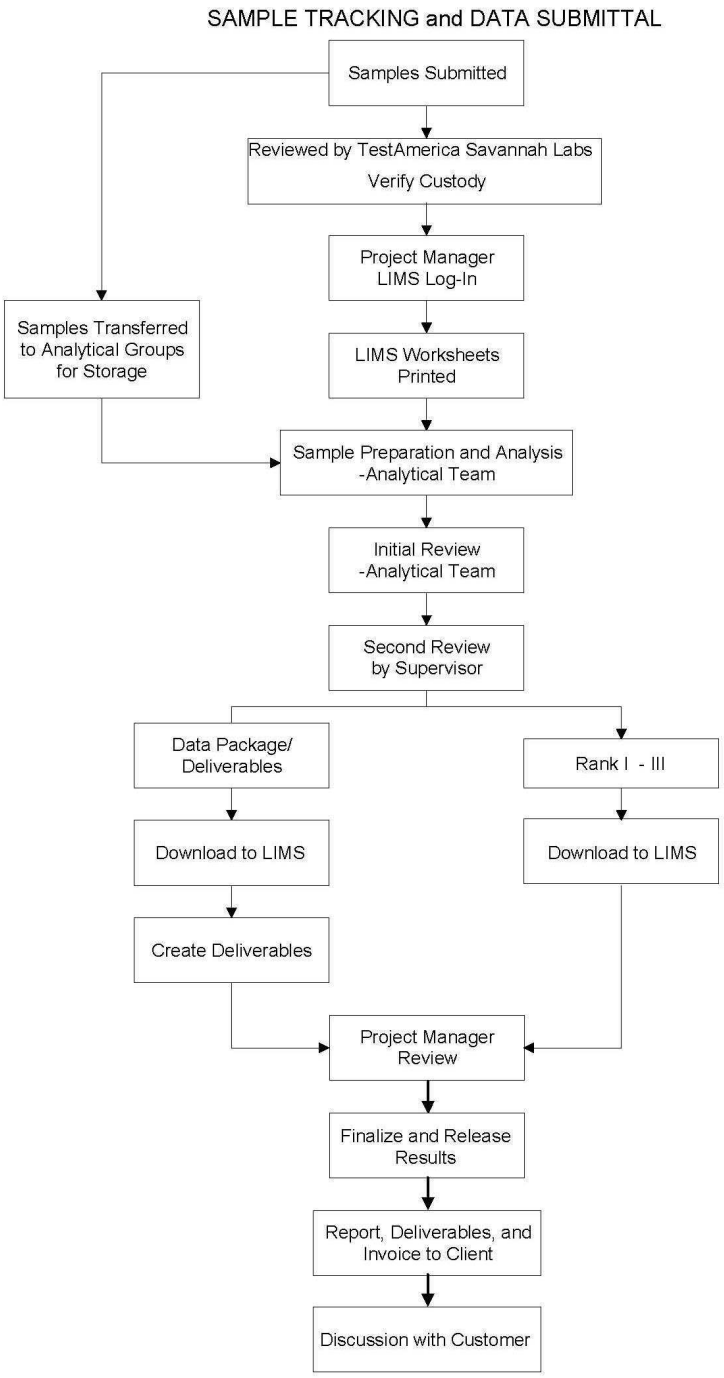
Figure 19-2. Work Flow



SECTION 20. EQUIPMENT AND CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the logbook used to monitor performance is also the maintenance logbook. Multiple pieces of equipment may share the same logbook as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logbooks are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logbooks shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control (e.g. CCV run on *'date'* was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to laboratory operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage, or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult the analytical SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer.

- If the temperature measuring device is used over a range of 10℃ or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10℃, then the verification must bracket the range of use.

IR thermometers, digital probes, and thermocouples are calibrated quarterly. IR Thermometers should be calibrated over the full range of use, including ambient and iced (4 degrees) per the EPA Drinking Water Manual.

The mercury NIST thermometer is recalibrated every three years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented electronically. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in equipment-specific logbooks or TALS sample batches. More information on this subject can be found in SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day – including weekends and holidays (i.e., 7 days a week).

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented on the Daily Temperature Log and in procedure-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Glass micro-syringes are considered the same as Class A glassware provided they are purchased with a manufacturer's certificate attesting to their accuracy. Micro-syringes are routinely purchased from Hamilton Company. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

Any device not regularly verified can not be used for any quantitative measurements.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration).

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed, if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules are standard ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or a vendor-certified different lot if a second source is not available). For unique situations, where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used, then bracketing calibration verification standards are not required, only daily verifications are needed, unless specified by the reference method. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements. Refer to the specific SOPs for requirements. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument/method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client:

a) when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b) when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated, and accepted.

Samples reported by the two conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

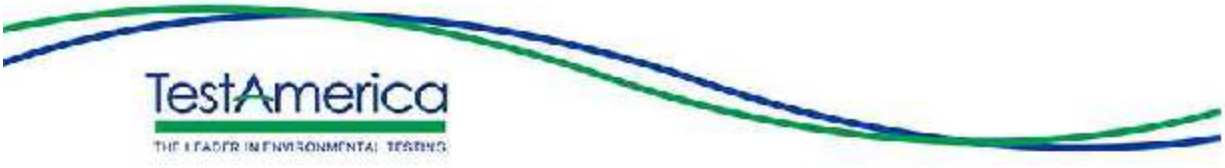
For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS Tuning

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Instrumentation



TestAmerica Savannah Instrument List

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
ICP	Varian (ICP E)	730-ES	IP0712M054	2008	New
ICP	Varian (ICP F)	730-ES	1P0803M118	2012	Used
ICP/MS	Agilent (ICP/MS B)	Agilent 7500CE G3272A	JP 14101289	2005	New
ICP/MS	Agilent (ICP/MS C)	Agilent 7700x G3281A	JP10390615	2011	New
CVAA	Leeman (2)	HYDRA AA II	00024	2011	New
GC/MS Semivolatiles	Hewlett-Packard (CMS D)	5973/6890	US82311451	1999	New
GC/MS Semivolatiles	Hewlett-Packard (CMS E)	5973/6890	US82311455	1999	New
GC/MS Semivolatiles	Hewlett-Packard (CMS F)	5973/6890	US44647039	2004	New
GC/MS Semivolatiles	Hewlett-Packard (CMS G)	5973/6890	US82311571	1999	New
GC/MS Semivolatiles	Hewlett-Packard (CMS K)	5973/6890	CN10524062	2005	New
GC/MS Semivolatiles	Hewlett-Packard CMS (N)	5973/6890	US72010580	1998	New
GC/MS Semivolatiles	Hewlett-Packard (CMS R)	5973/6890N	21842170	2002	New
GC/MS Semivolatiles	Hewlett-Packard (CMS T)	5973/6890	US33246115	2003	New
GC/MS Semivolatiles	Agilent (CMS W)	5975/6890N	US10608004	2006	New
GC/MS Semivolatiles	Hewlett-Packard (CMS X)	5975/6890N	CN10608061	2006	New
GC/MS Semivolatiles	Agilent (CMS Y)	5975/7980A	US80838915	2008	New
GC/MS Semivolatiles	Hewlett-Packard (CMSAE)	5973/6890	US82311503	2015	Used
GC/MS Volatiles	Hewlett-Packard (CMS A)	5973/6890	US82311453	2000	New
GC/MS Volatiles	Hewlett-Packard (CMS AA)	5973/6890N	US406220567	2013	Used

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GC/MS Volatiles	Hewlett-Packard (CMS B)	5973/6890	US82311452	2000	New
GC/MS Volatiles	Hewlett-Packard (CMS C)	5975/7890	CN10917056	2012	New
GC/MS Volatiles	Hewlett-Packard (CMS O)	5973/6890	US7200579	1993	New
GC/MS Volatiles	Hewlett-Packard (CMS P)	5973/6890	US0039011	2000	New
GC/MS Volatiles	Hewlett-Packard (CMS S)	5973/6890	US21843181	2002	New
GC/MS Volatiles	Agilent (CMS U)	5973/6890	US52441057	2005	New
GC/MS Volatiles	Agilent (CMSAC)	5973/6890	US82311488	2014	Transfer
GC/MS Volatiles	Agilent (CMSAD)	5973/6890	US82311485	2014	Transfer
Ion Chromatograph	Dionex (CIC N)	ICS-1000	04060105		Used
Ion Chromatograph	Dionex (CIC G)	ICS-2000	05101132	2005	New
Ion Chromatograph	Dionex (CIC H)	ICS-2000	06080799	2006	New
Ion Chromatograph	Dionex (CIC K)	ICS-2000	0307011	2012	Used
Ion Chromatograph	Dionex (CIC L)	ICS-2000	05120486	2012	Used
GC Semivolatiles	Hewlett-Packard (CSG J)	6890 (ECD)	US00033184	2000	New
GC Semivolatiles	Hewlett-Packard (CSG K)	6890 (ECD)	US10223085	2002	New
GC Semivolatiles	Agilent (CSG Q)	6890N (FID)	CN10521056	2005	New
GC Semivolatiles	Hewlett-Packard (CSG S)	6890 Plus (ECD)	US00024188	2000	New
GC Semivolatiles	Hewlett-Packard (CSG X)	6890N (ECD)	CN10406086	2003	New
GC Semivolatiles	Agilent (CSG Y)	6890N (ECD)	CN10528081	2005	New
GC Semivolatiles	Agilent (CSG Z)	6890N (ECD)	CN10814004	2008	New
GC Semivolatiles	Agilent (CSGAA)	6890 (ECD)	US00031692	2013	Used
GC Semivolatiles	Agilent (CSG AB)	6890N(FID)	US10224026	2013	Used
GC Semivolatiles	Agilent (CSG AD)	6890N (ECD)	4510326002	2015	Used
GC Volatiles	Agilent (CVG G)	6890 (FID)	14921	2007	New
GC Volatiles	Agilent (CVG U)	6890 (FID)	US10439011	2005	New

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GC Volatiles	Agilent (CVG V)	6890 (FID)	CN10619098	2006	New
GC Volatiles	Agilent (CVG W)	6890 (FID)	CN10603131	2006	New
Liquid Chromatography	Hewlett-Packard (CLC J)	1100	US72101013	2002	New
Liquid Chromatography	Hewlett-Packard (CLC K)	1100	US72102590	2002	New
Liquid Chromatography	Hewlett-Packard (CLC N)	1100	DE91607527	2008	Used
General Chemistry	Hach (TURB1)	2100 AN	950400000487	1995	New
General Chemistry	Lachat (1)	QuickChem 8000	A83000-1070	1997	New
General Chemistry	Lachat (2)	QC 8500 Series 2	100200001169	2010	New
General Chemistry	Lachat (3)	QuickChem 8000	A8300-1086	2012	Used
General Chemistry	BOD AssayPlus	Version 3.0	270F6XB334	2006	New
General Chemistry	PCTitrate	Version 3.0	270G6XB370	2006	New
General Chemistry	Konelab (1)	Konelab20	M4218134	2000	New
General Chemistry	Konelab (2)	Konelab20	M3118114	2001	New
General Chemistry	Konelab (3)	Konelab20	S2519236	2013	Used
General Chemistry	Analyik jena	Multi X 2500	N1-197/O	2014	New
General Chemistry	Shimadzu	TOC-L	H54335232053	2015	New
General Chemistry	Hach (SPC7)	DR2800	1359509	2016	Used
Extractions	Hach (SP6)	DR2800	1342967	2016	Used

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, deionized (DI) water systems, automatic pipettes, and other volumetric measuring devices. (Refer to Section 20.3.) With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices (daily for DOD). Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A glassware and glass microliter syringes should be routinely inspected for chips, acid etching, or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia-Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

A calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty. The calibration report or certificate contains a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. All calibration reports are filed in the QA Department.

An external certified service provider services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. Mercury thermometers are verified annually against a traceable reference thermometer. Digital thermometers are verified quarterly against a traceable reference thermometer. Temperature readings of ovens, refrigerators, freezers, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique reagent ID and expiration date. All documentation received with the reference standard is retained as a QC record and references the reagent ID.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be, at a minimum, the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. (Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.)

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained electronically. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's TALS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the TALS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- TALS Standard ID
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained electronically.

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container
- Recommended Storage Conditions

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets, and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

TestAmerica Savannah provides limited sampling services. Sampling procedures are described in SOP SA-FD-05: *Field Sampling Procedures.*

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness provided by the supplier are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote, or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need

consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples.*

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time, and location of sampling
- Sample collector's name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of

the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (e.g., Fed-Ex, UPS), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date as it lists all receipts on each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

Additional information on the sample receipt process is given in SOP SA-CU-01: *Sample Receipt Procedures.*

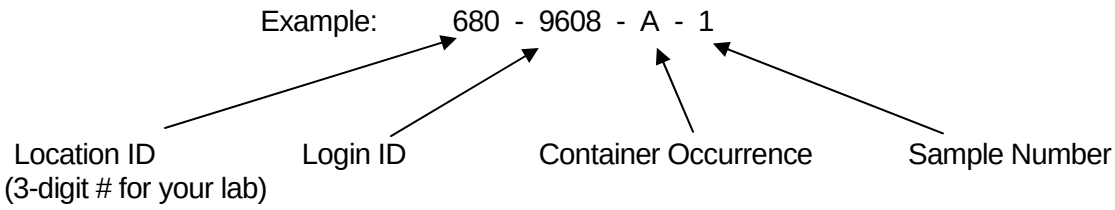
23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on the Sample Receipt Checklist in TALS and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at any time. This system includes identification for all samples, subsamples, and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Savannah is the laboratory (Location ID 680). The Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 680 - 9608 - A - 1 - **A** ←——**Secondary Container Occurrence**

Example: 680-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the Project Manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and route them to the appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

23.4 Sample Storage

In order to avoid deterioration, contamination, or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers, or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, storage blanks are maintained in the volatile sample refrigerators and analyzed every week.

Analysts retrieve the sample container allocated for their analysis from the designated storage location, prepare or analyze the sample, and return the remaining sample to the storage location from which it originally came. All samples are scanned into and out of the storage locations using the TALS sample custody program. Empty containers are scanned into the TALS sample custody program as empty and are properly disposed of. All samples are kept for at least 30 days after the report is sent out, which meets or exceeds most sample holding times. After this time, the samples are properly disposed of in accordance with the Environmental Health and Safety Manual.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples and Foreign Soils

Upon receipt, foreign soil samples are marked with a fluorescent green “FOREIGN SOIL” label prior to distributing to the analytical departments. Once the sample is received by the department, it is stored in a “FOREIGN SOIL ONLY” box segregated from other samples. Non-hazardous foreign soil samples are sent out for incineration by a USDA-approved waste

disposal facility. RCRA hazardous foreign soil samples are heat treated at the laboratory. After heat treatment, normal disposal procedures are followed. Refer to the Environmental Health and Safety Manual Addendum for additional information on disposal of hazardous samples. If not classified as hazardous, foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they did not inadvertently omit a key part of regulatory compliance testing.

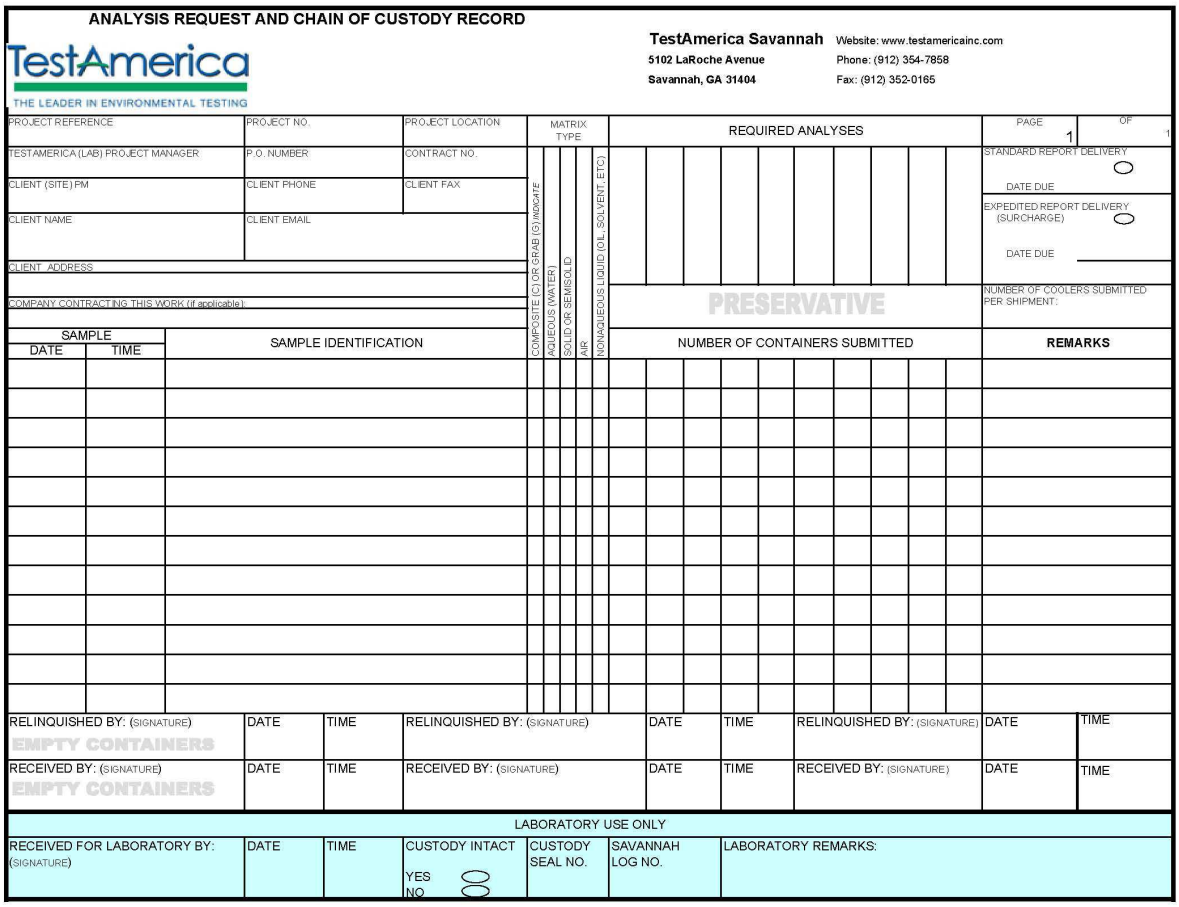
23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures outlined in the Savannah Addendum to the Environmental Health and Safety Manual. All procedures in the laboratory Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than three months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or

deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Chain of Custody (COC)



ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Savannah Website: www.testamericainc.com
5102 LaRoche Avenue Phone: (912) 354-7858
Savannah, GA 31404 Fax: (912) 352-0165

PROJECT REFERENCE	PROJECT NO.	PROJECT LOCATION
TESTAMERICA (LAB) PROJECT MANAGER	P.O. NUMBER	CONTRACT NO.
CLIENT (SITE) PM	CLIENT PHONE	CLIENT FAX
CLIENT NAME	CLIENT EMAIL	
CLIENT ADDRESS		
COMPANY CONTRACTING THIS WORK (if applicable):		

MATRIX TYPE: COMPOSITE (C) OR GRAB (G) INDICATE; AQUEOUS (WATER); SOLID OR SEMISOLID; AIR; NONAQUEOUS LIQUID (OIL, SOLVENT, ETC)

REQUIRED ANALYSES

PAGE 1 OF 1

STANDARD REPORT DELIVERY ⬭
DATE DUE
EXPEDITED REPORT DELIVERY (SURCHARGE) ⬭
DATE DUE

PRESERVATIVE

NUMBER OF COOLERS SUBMITTED PER SHIPMENT:

SAMPLE DATE	SAMPLE TIME	SAMPLE IDENTIFICATION	NUMBER OF CONTAINERS SUBMITTED	REMARKS

RELINQUISHED BY: (SIGNATURE) EMPTY CONTAINERS	DATE	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME
RECEIVED BY: (SIGNATURE) EMPTY CONTAINERS	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME

LABORATORY USE ONLY

RECEIVED FOR LABORATORY BY: (SIGNATURE)	DATE	TIME	CUSTODY INTACT YES ⬭ NO ⬭	CUSTODY SEAL NO.	SAVANNAH LOG NO.	LABORATORY REMARKS:

FCU052:01.02.08:2

Figure 23-2. Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax, or e-mail ASAP after the receipt of the samples.

1) Samples must arrive in good condition with a Chain-of-Custody (COC) filled out completely.

2) Samples must be properly labeled.

3) Samples must be in proper containers with adequate volume for the analysis. Aqueous samples submitted for Volatiles analyses must be submitted without headspace. Samples must be dechlorinated and submitted with proper chemical preservation (pH) as required by the analytical test method.

4) Most analytical methods require chilling samples to 4^{o}C. These criteria are met if the samples are chilled to below 6^{o}C and above freezing. Note: Samples hand-delivered to the laboratory immediately after collection are only considered acceptable if there is evidence that the chilling process has begun (i.e., arrival on ice).

5) Samples must be prepared and analyzed with the holding times defined in the analytical test method.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

Figure 23-3. Login Sample Receipt Checklist

Login Sample Receipt Check List

Client: TestAmerica Laboratories, Inc.

Job Number:

SDG Number:

Login Number:

List Source:

Creator:

List Number:

Question	T / F/ NA	Comment
Radioactivity either was not measured or, if measured, is at or below background		
The cooler's custody seal, if present, is intact.		
The cooler or samples do not appear to have been compromised or tampered with.		
Samples were received on ice.		
Cooler Temperature is acceptable.		
Cooler Temperature is recorded.		
COC is present.		
COC is filled out in ink and legible.		
COC is filled out with all pertinent information.		
There are no discrepancies between the sample IDs on the containers and the COC.		
Samples are received within Holding Time.		
Sample containers have legible labels.		
Containers are not broken or leaking.		
Sample collection date/times are provided.		
Appropriate sample containers are used.		
Sample bottles are completely filled.		
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs		
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.		
If necessary, staff have been informed of any short hold time or quick TAT needs		
Multiphasic samples are not present.		
Samples do not require splitting or compositing.		
Is the Field Sampler's name present on COC?		
Sample Preservation Verified		

TestAmerica Savannah

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying, and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Control Type	Details
Method Blank (MB)	Used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	Prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

Control Type	Details
Instrument Blanks	Blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	Required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	Sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	Sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Holding Blanks	Referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon method performance (e.g., Laboratory Control Sample), which entails both the preparation and measurement steps; and matrix effects (e.g., Matrix Spike or Sample Duplicate), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology, and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as phosphorus), a calibration verification standard is reported as the LCS.

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, Toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSDs are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS

and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or surrogate spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary (recommended on an annual basis) unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (CCV) unless the analytical method specifies a tighter limit.

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- For routine analytes that are not classified as poor performers, the lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable).

- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart may be visually inspected and, using professional judgment, they may be left unchanged if there is no effect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

The QA Department generates a Method Limit Group (MLG) in the TALS that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Savannah. The MLG includes an effective date and is updated each time new limits are generated and entered. Unless otherwise noted, limits within these tables are

laboratory generated. The TALS maintains an archive of all limits used within the laboratory.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- If there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

Marginal exceedances should be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is reviewed and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report) with a "Result" column header.

25.2.2 Each report cover page includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. Job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Reporting limits

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.19 When TNI accreditation is required, the laboratory shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.20 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.21 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.22 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.23 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or preliminary report). A complete report must be sent once all of the work has been completed.

25.2.24 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.25 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above, excluding QC data.
- Level II is a Level I report plus summary information, including QC results.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. TestAmerica Savannah offers a variety of EDD formats including Environmental Resources Program Information Management System (ERPIMS), Automated Data Review (ADR), Locus Focus (EIM), EQUIS ESBasic, Environmental Quality Information Systems (EQUIS), Staged Electronic Data Deliverable (SEDD), EPA Region V EDD (EDMAN), and Terrabase.

EDD specifications are submitted to the IT department by the Project Manager for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without

errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify sender immediately.

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained in the TALS, as is the original report. The revised report is stored in the project files under the sample number followed by "Rev#" where # is the number of the report revision.

When the report is re-issued, the revision number is placed on the cover/signature page of the report or at the top of the narrative page. A brief explanation of reason for the re-issue and a reference back to the last final report generated may be included.

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

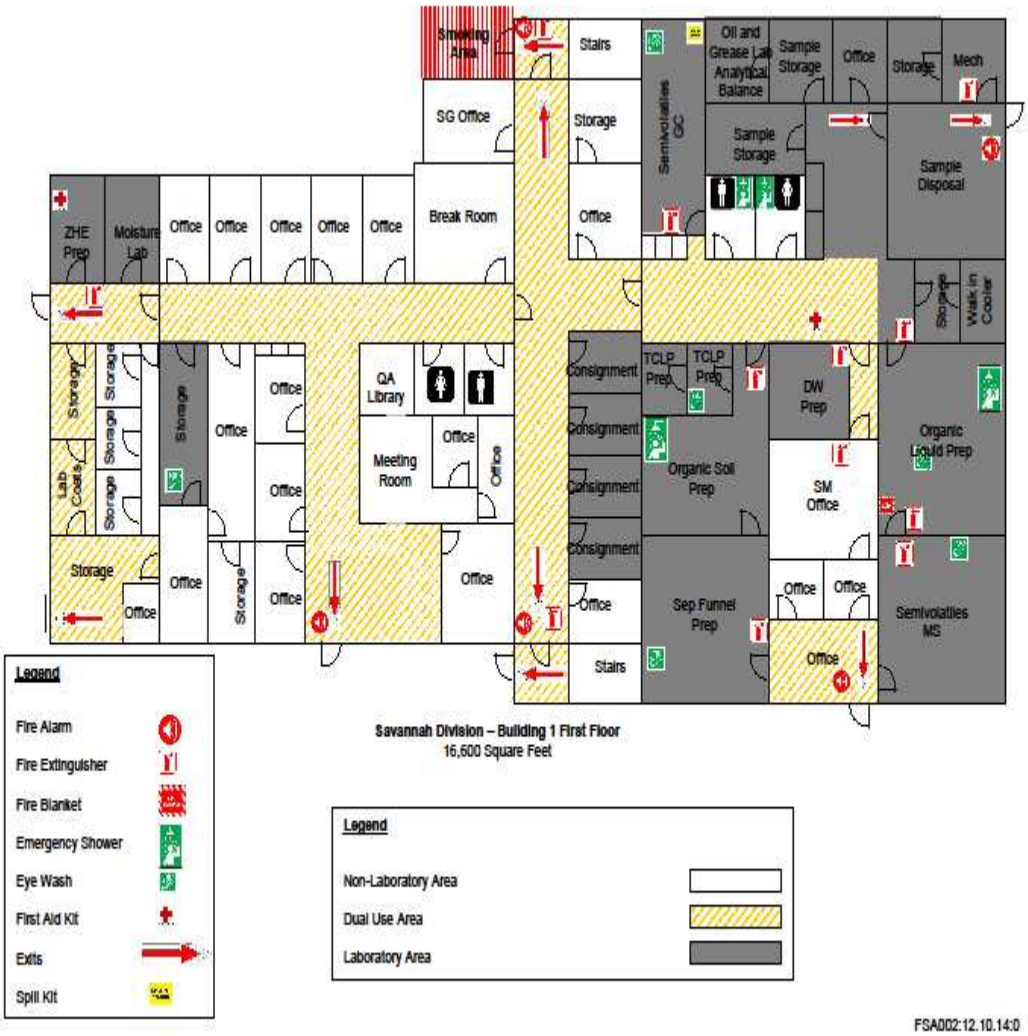
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

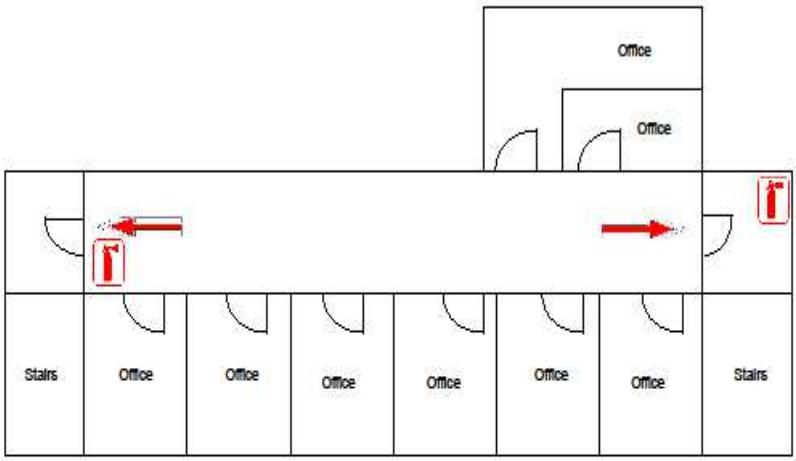
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists EPA Method 8315 but client wanted EPA Method 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. Laboratory Floor Plan





Legend

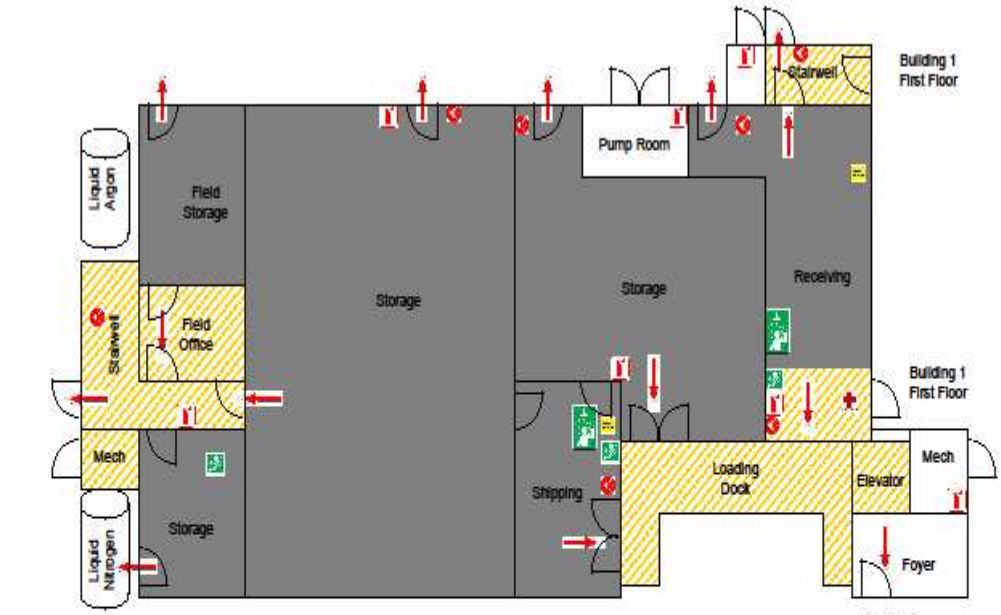
Fire Alarm
Fire Extinguisher
Fire Blanket
Emergency Shower
Eye Wash
First Aid Kit
Exits
Spill Kit

Savannah Division – Building 1 Second Floor
2,200 Square Feet

Legend

Non-Laboratory Area
Dual Use Area
Laboratory Area

FSA003:12.10.14:0



Savannah Division – Building 2 First Floor
8,400 Square Feet

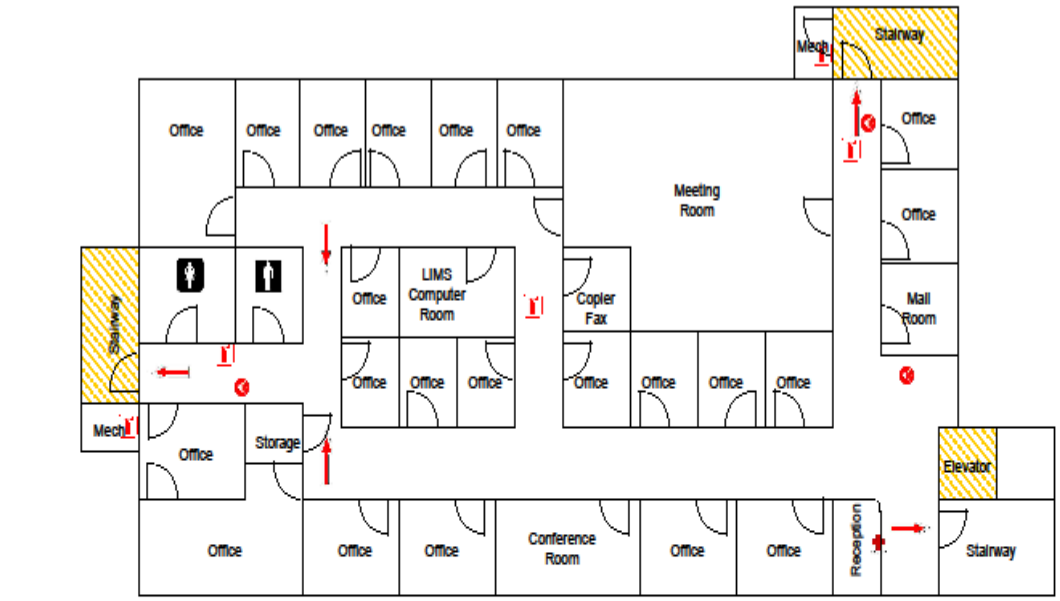
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA004:12.10.14:0



Legend

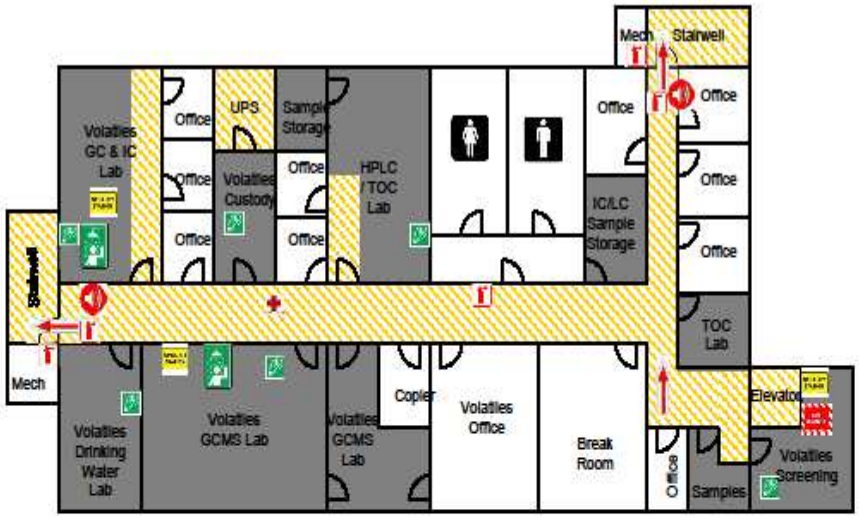
- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Savannah Division – Building 2 Second Floor
8,400 Square Feet

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA005:12.10.14:0



Savannah Division – Building 2 Third Floor
8,400 Square Feet

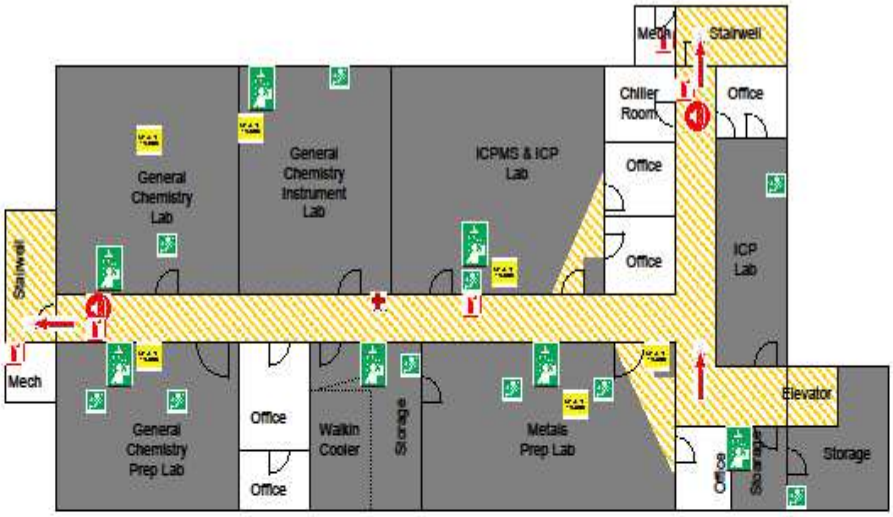
Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA006:11.23.15:1



Savannah Division – Building 2 Fourth Floor
8,400 Square Feet

Legend

- Fire Alarm
- Fire Extinguisher
- Fire Blanket
- Emergency Shower
- Eye Wash
- First Aid Kit
- Exits
- Spill Kit

Legend

- Non-Laboratory Area
- Dual Use Area
- Laboratory Area

FSA007:12.10.14:0

Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

Glossary:

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:
The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (TNI)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (TNI)

Accuracy:
The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Aliquot:
A representative portion of the sample, standard, or reagent.

Analyst:
The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

Analyte:
The element, molecule, or compound that is being measured in a given procedure. Also referred to as a parameter.

Analytical Method:
Defines the sample preparation and instrumentation procedures that must be performed to determine the quantity of analyte in a sample.

Analytical Sequence:
The order in which calibration standards, verification standards, QC items, and samples are analyzed.

Analytical Spike:
Addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anion:
A negatively charged ion.

Anomaly:
Anomalous situations that are out of the ordinary but are not necessarily a method deviation and are not definitive enough to require a CAR are documented in the Non-Conformance Module. The use of the grand mean exception would require initiation of an Anomaly NCM.

Aromatic:
Relating to the six-carbon-ring configuration of benzene and its derivatives.

Assessment:
The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Assessment Team:
The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (TNI)

Assessor:
One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Background Correction:
A technique to compensate for variable background contribution to the instrument signal and the determination of trace metals.

Batch:
Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias:
The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank:
A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Blind Sample:
A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (TNI)

Calibration Check Compounds (CCC):
Term used in conjunction with SW-846, Method 8260 and 8270 to refer to the compounds in which the percent RSD is evaluated against method-prescribed criteria to decide the validity of a calibration.

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Method:
A defined technical procedure for performing a calibration. (TNI)

Calibration Standard:
A substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain of Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (TNI)

Cation:
A positively charged ion.

Chemical Analysis:
Any of a variety of laboratory methods used to evaluate the concentrations of compounds and elements present in an environmental sample.

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them.

Client Complaint:
A complaint is a situation where dissatisfaction is expressed with the service provided by the laboratory.

Composite Sample:
Portions of material collected from more than one spatial location or at different times that are blended and submitted for chemical analyses. Composite samples can provide data representative of a large area with relatively few samples. However, the resulting data are less accurate with regard to the concentrations of contaminants detected in a specific location, because they represent average values.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites.

Compromised Samples:
Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (TNI)

Concentration:
The mass of analyte per unit mass or volume of sample. Common units of concentration for environmental analyses are microgram per liter or kilogram (ug/L or ug/kg) and milligrams per liter or kilogram (mg/L or mg/kg).

Confidence interval:
For normally distributed (random) data, the intervals where 68%, 95%, and 99% of the data fall. 68% of the data should fall within 1 standard deviation of the mean, 95% of the data should fall within 2 standard deviations of the mean, and 99% of the data should fall within 3 standard deviations of the mean.

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures
(TNI)

Conformance:
An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Continuing Calibration Verification (CCV) Standard:
A mid-concentration analytical standard run periodically to verify the calibration of the analytical instrument. Also known as continuing calibration check (CCC).

Contract Laboratory Program (CLP):
A nationwide laboratory network established by the USEPA, structured to provide legally defensible analytical results to support USEPA enforcement actions or other requirements of the use community. The CLP incorporates a level of quality assurance appropriately designed for the intended usage of the data.

Control Limits:
Accuracy or precision ranges that determine whether the experimentally determined results are in control. If the results are within the acceptance ranges, the results are said to be in control; if the results are outside the limits, they are said to be out-of–control.

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Corrective Action Report (CAR):
The CAR form is used in situations where a recurring problem or breakdown in systems is observed and warrants a more thorough investigation than a single-event NCR. CARs may be initiated from: a specific nonconformance situation (NCM), an observed trend or frequency of events that warrant corrective action, an audit finding, etc.

Correlation Coefficient:
A number (r), which indicates the degree of dependence between two variables (concentration and response). The more dependent the variables are, the closer the value is to one. This value is used to evaluate the straightness of a line, (the linearity of the instrument).

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (TNI)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Data Validation:
An evaluation of laboratory data quality based on a review of the data deliverables. This process involves procedures verifying instrument calibration, calibration verification, and other method-specific performance criterion.

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability (DOC):
Procedure to establish the ability to generate acceptable accuracy and precision. This is done initially upon starting a new method and then continues each year the method is performed.

Detection Limit:
The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (TNI)

Direct Aqueous Injection (DAI):
A technique in which an aliquot of the aqueous sample or aqueous leachate is injected directly into the gas chromatograph with no prior sample preparation.

Disposal:
Final placement or destruction of wastes. Disposal may be accomplished through the use of landfills, treatment processes, etc.

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:
The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

E. coli:
Bacteria giving a positive total coliform response and possessing the enzyme B-glucuronidase, which cleaves the fluorogenic substrate MUG, resulting in the release of a fluorescent product when viewed under long-wavelength UV light.

Environmental Detection Limit (EDL):
The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (TNI Radioanalysis Subcommittee)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Extractable Organics:
Semivolatiles (base/neutral and acid extractable compounds) and pesticide/polychlorinated biphenyl compounds that can be partitioned into an organic solvent from the sample matrix and are amenable to gas chromatography (GC).

Fecal Coliforms:
A subset of total coliforms that grow and ferment lactose at an elevated incubation temperature (44.5℃) and are also referred to as thermotolerant coliforms. Fecal coliforms produce colonies that appear in various shades of blue, domes and glistening, ranging in size from pinpoints to several millimeters. This group consists of mostly E. Coli (EC) but also includes some other enterics. Fecal coliforms are a more specific indicator organism for contamination. This type of bacteria is associated with the fecal material of warm-blooded animals.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (TNI)

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field Control Samples:
General term assigned to field-generated replicates (duplicates/splits/spikes), blanks, background/upgradient samples, etc.

Field Duplicate Sample:
Independent sample collected at approximately the same time and place, using the same methods as another sample. The duplicate and original sample are containerized, handled, and analyzed in an identical manner.

Field of Testing:
TNI's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (TNI)

Filtrate:
A filtered liquid.

Filtration:
The physical removal of solid particles from a liquid wastestream by passing the liquid across a filter medium, which serves as a barrier to the solid material.

Finding:
An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (TNI)

Gas Chromatography/Mass Spectroscopy (GC/MS):
Two distinct analytical techniques used to separate and identify organic compounds: the GC is used for the separating portion and the MS is used as the detection portion of an analysis. Both techniques are typically performed by a single instrument.

Good Laboratory Practices (GLP):
Formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729.

Heavy Metals:
In reference to environmental sampling, typically identified as the following trace inorganics: cadmium, lead, mercury, silver, etc. (all metals of health concern). Heavy metals can cause biological damage if consumed at low concentrations and tend to accumulate in the food chain.

Heterotrophic Bacteria:
A large group of bacteria that obtain energy by oxidizing organic matter. Coliform bacteria are a subset of this group.

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Homogeneous:
The quality of uniform composition.

Initial Calibration Verification (ICV):
A mid-concentration analytical standard run immediately after the calibration to verify the calibration of the analytical instrument. Also known as initial calibration check (ICC).

Inorganic Chemicals:
Chemical substances of mineral origin, not of basically carbon structure.

Inquiry:
A question or request for information about the service provided by the laboratory.

Inspection:
An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Instrument Blank:
A blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Instrument Detection Limit (IDL):
The minimum amount of a substance that can be measured on a specific instrument, with a specified degree of confidence that the amount is greater than zero. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. An IDL value, by definition, has an uncertainty of ±100%. The IDL thus represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Instrument Performance Check Solution (IPC):
A solution of one or more method analytes, surrogates, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

Intermediate or Secondary Stock Standard:
A solution made from two or more stock standards. A secondary standard may also be a certified solution purchased from a vendor as a mixture of several target analytes. Also known as a source reagent in TALS if purchased and an intermediate reagent if prepared in the lab.

Internal Standard:
A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration:
Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:
A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Response:
Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

Job number:
A sequential number that is assigned to each client's samples upon receipt into the laboratory. This log number provides the primary means of associating the samples to the client.

Laboratory:
A defined facility performing environmental analyses in a controlled and scientific manner. (TNI)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (TNI)

Laboratory Duplicate:
Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (TNI)

Laboratory Fortified Blank (LFB):
An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements at the required method detection limit. The percent recovery (accuracy) result for the LFB must fall within the limits listed in the TALS. Also referred to as a laboratory control standard (LCS).

Laboratory Fortified Sample Matrix (LFM):
An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations. The percent recovery (accuracy) result for the LFM must fall within the limits listed in the TALS. Also referred to as a matrix spike (MS).

Laboratory Fortified Sample Matrix Duplicate (LFMD):
A replicate laboratory fortified sample matrix.

Laboratory Performance Check Solution (LPC):
A solution of selected method analytes used to evaluate the performance of the instrumental system with respect to a defined set of method criteria.

Laboratory Quality Manual (LQM):
A document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system. Also referred to as the Quality Assurance Manual (QAM) or Quality Assurance Plan (QAP).

Laboratory Reagent Blank (LRB):
An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Also referred to as a method blank (MB).

Leachate:
The liquid portion of a sample that passes through a 0.6µm filter in the initial evaluation of the percent solids, or the liquid that passes through a 0.6µm filter after the sample has been subjected to the TCLP. The liquid produced by subjecting the sample to the SPLP method.

Least Squares Regression (1^{st} Order Curve):
The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):
An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Liquid phase:
The portion of the sample that passes through the 0.6-0.8□m filter when subjected to a pressure of 50psi during the TCLP or SPLP process.

Manager (however named):
The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (TNI)

Mass Spectrometry (MS):
A detection instrument that differentiates compounds by their differences in mass, or mass fragments. The basic components of the MS are the ion source and lenses, the mass filter (quadrapoles), and the electron multiplier. The ion source and lenses create the ions and propel them on a consistent path to the quadrapoles. The quadrapoles filter the ions that are produced in the source, allowing them to continue to the electron multiplier, where the ions are collected and the signal sent to the data system.

Mass Spectra:
A graphical representation of the abundance of the mass ions produced when a compound is detected by mass spectrometry. The mass spectrum is essentially a fingerprint of the compound and along with the retention time of the compound provides excellent qualitative information about the presence of the compound.

Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Duplicate (MD):
Duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (spiked sample or fortified sample):
Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:
A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (TNI)

Method Detection Limit:
The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)
The MDL is defined as:

$$MDL = SD \otimes t(0.99)$$

SD = standard deviation of the replicates
t(0.99) = Student's t-Value at the 99% confidence level for number of replicates

Most Probable Number (MPN):
An estimate of the mean density of coliforms in a sample based on certain probability formulas.

National Environmental Laboratory Accreditation Conference (NELAC):
A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (TNI)

National Environmental Laboratory Accreditation Program (NELAP):
The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (TNI)

Neat standard:
A pure compound, element, or salt that contains the target analyte. The purity, usually expressed as a percent, of the neat standard must be known. Also known as a source reagent in TALS.

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (TNI)

NELAC Standards:
The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (TNI)

Non-conformance:
Any occurrence that prevents the lab from delivering data that is compliant with the control criteria published (or incorporated by reference) in an applicable QA plan. The Non-conformance Module is used to document nonconformance conditions and to specify the necessary action(s) taken to correct the specific problem.

Organic:
Referring to or derived from living organisms; any compound containing carbon.

Parts Per Billion (ppb):
One part of analyte per billion parts of sample. For aqueous samples, a ppb is equivalent to ug/L; for soils, ug/kg.

Parts Per Million (ppm):
One part of analyte per million parts of sample. For aqueous samples, a ppm is equivalent to mg/L; for soils, mg/kg.

Peak Gaussian Factor (PGF):
A means to measure peak symmetry and monitoring retention time drift over time. Critically evaluate peak in the instrument performance check sample, and calculate the PGF as follows,

$$PGF = \frac{1.83 \otimes W(1/2)}{W(1/10)}$$

where:
W(1/2) is the peak width at half height
W(1/10) is the peak width at tenth height

Percent Recovery:
Percent recovery is used to assess accuracy and is calculated:

$$\%REC = \frac{C\,\text{exp}\,erimental}{Cknown} \otimes 100$$

where:
$C_{experimental}$ = experimentally determined concentration
C_{known} = known or theoretical concentration

Percent Solids:
The proportion of solid in a soil sample determined by drying an aliquot of the sample.

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (TNI)

Performance Based Measurement System (PBMS):
A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (TNI)

pH:
A numerical designation of relative acidity or basicity (Alkalinity). A pH of 7 indicates neutrality; lower values indicate increasing acidity; high values indicate increasing alkalinity.

Precision:
The agreement between two or more experimentally determined results. Precision is routinely expressed as the relative percent difference between two results. Precision is not routinely used as a measurement to determine if the analysis is in control but may be required for certain programs and agencies.

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (TNI)

Post-Digestion Spike:
Addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed.

Preservation:
Refrigeration and or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample. Methods used to retard degradation of chemical analytes within samples by inhibiting decomposition by biological action, chemical reactions, and reducing sorption effects. Methods include limiting headspace, chemical, acid, or base addition, protection from light, cooling, etc.

Precision:
The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation:
Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (TNI)

Preventive Action:
The pro-active process of noting and correcting a potential problem before it happens due to a weakness in a system, method, or procedure.

Procedural Standard Calibration:
A calibration method where aqueous calibration standards are prepared and processed (e.g., purged, extracted, and/or derivatized) in exactly the same manner as a sample. All steps in the process from addition of sampling preservatives through instrumental analyses are included in the calibration. Using procedural standard calibration compensates for any inefficiency in the processing procedure.

Proficiency Testing:
A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Quality Assurance:
An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual:
A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System:
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

Quantitation Limit (QL):
The lowest point at which a substance can be quantitatively measured with a specified degree of confidence using a specific method. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL) or Reporting Limit (RL).

Quantitation Limits:
The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (TNI)

Range:
The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data:
Any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports

specifying inclusion of “raw data” do not need all of the above included, but sufficient information to create the reported data.

Reagent:
A material that is used in a process or analysis but is not directly related to the measured analyte concentration.

Reagent Blank (method reagent blank):
A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:
A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (TNI)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Regulatory Threshold Limit:
The concentration of analyte in the TCLP leachate at which the sample is deemed hazardous.

Relative Percent Difference:
The relative percent difference is calculated between the concentrations of two spikes or sample duplicates:

$$\%RPD = \left| \frac{(C1 - C2)}{\frac{C1 + C2}{2}} \right| \otimes 100$$

Where:
C_1 = concentration of the sample or spike
C_2 = concentration of the sample duplicate or spike duplicate

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (TNI)

Reporting Limit (RL):
Defines the lowest concentration that can be reported with reasonable certainty that the result falls within the laboratories’ accuracy and precision limits. Also referred to as the practical quantitation limit or PQL, the RL is usually defined as the lowest point in the calibration curve or the sample equivalent concentration of the lowest point in the calibration curve.

Representativeness:
A qualitative measure of the extent to which a sample(s) acquired from a medium describes the chemical characteristics of that medium.

Requirement:
Denotes a mandatory specification; often designated by the term “shall”. (TNI)

Resolution:
Also known as separation, or percent resolution. The separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smallest peak being resolved, and multiplied by 100.

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the “cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal. (TNI)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (TNI)

Sample:
A portion of material collected for chemical analyses. Note that a sample is identified by a unique sample number and that the term and the number may apply to multiple sample containers, if a single sample is submitted for a variety of chemical analyses.

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Sampling and Analysis Plan (SAP):
A formal document describing the detailed sampling and analysis procedures for a specific project.

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.990.

Secondary or Intermediate Stock Standard:
A solution made from two or more stock standards. A secondary standard may also be a certified solution purchased from a vendor as a mixture of several target analytes. Also known as a source reagent in TALS if purchased and an intermediate reagent if prepared in the lab.

Selectivity:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Semivolatile Organics:
Compounds that are amenable to analysis by extraction of the sample with an organic solvent. The term semivolatile organic is used synonymously with base/neutral/acid (BNA) compounds.

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Solvent:
The organic liquid used to extract the compounds of interest out of the sample matrix. The solvent is also used to dissolve (put into solution) standards. In general, the solvent used to prepare the standards is also used to extract the samples. A good rule of thumb is that “like dissolves like”, that is, a solvent must be similar in chemical structure to the compound that is being extracted or being dissolved. For most organic extractions, the solvent should also not be miscible (dissolves in all proportions) with water.

Spike:
A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period. (TNI)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of TNI and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):
A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Stock standard:
A solution made from one or more neat standards. The stock standard will usually have a high concentration, usually higher than 1000mg/L (1000ug/mL). This standard can also be purchased from a certified vendor. Also known as a source reagent in TALS.

Storage Blank:
A blank matrix stored with field samples of a similar matrix.

Supervisor (however named):
The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (TNI)

Surrogate:
A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Suspended Metals:
The concentration of metals determined in the portion of a sample that is retained on a 0.45-µm filter. (The concentration of suspended metals may also be calculated from the difference between the total metals sample results minus the dissolved metals sample results.)

Systems Audit (also Technical Systems Audit):
A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

System Performance Check Compounds (SPCCs):
Term used in conjunction with SW-846, Method 8260 and 8270, to refer to the compounds in which the response factor (RF) is evaluated against method-prescribed criteria to decide the validity of a calibration.

Target Analyte List (TAL):
Refers to the Contract Lab Program (CLP) list of inorganic analytes that includes metals and cyanide. May also refer to any general list of inorganic target analytes.

Target Compound List (TCL):
Refers to the Contract Lab Program (CLP) list of organic compounds that includes volatiles (GC/MS), semivolatiles (GC/MS), and pesticides and PCBs (GC/EC). May also refer to any general list of organic target compounds.

Technical Director:
Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (TNI)

Test:
A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:
An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (TNI)

Total Coliforms:
Gram-negative, facultative anaerobic rod-shaped enteric bacteria that ferment lactose to produce colonies with a metallic sheen (yellow to green) when viewed under a fluorescent lamp or acid and gas within 48 hours incubated at 35℃. All bacteria possessing the enzyme B-D-galactosidase, which cleaves the chromogenic substrate ONPG, resulting in release of a chromogen that produces a color change in the sample. They are used as an indicator of contamination in samples although some total coliform bacteria are found naturally in environmental samples. This type of bacteria is commonly found in the intestines of humans.

Total Metals:
Concentration of metals determined in an unfiltered water sample which is preserved (acidified) in the field, transported to the laboratory, and then follows a rigorous digestion.

Total Recoverable Metals:
Concentration of metals in an unfiltered water sample which is preserved (acidified) in the field and transported to the lab, which then performs the digestion with hot dilute mineral acid. This preparation method is typically utilized for drinking water samples and TCLP extracts.

Toxic Substances Control Act (TSCA):
The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (TNI)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Trip Blank:
Samples prepared by adding clean, analyte-free water to sample containers for analysis for volatile organics. Preservatives are added to the blank, and the containers are sealed prior to the sampling trip. Trip blanks are transported with empty sample containers to the site of work and remain sealed until analyzed with collected environmental samples. Trip blanks permit evaluation of contamination generated from sample containers or occurring during the shipping and laboratory storage process.

Tune:
To adjust the parameters of the mass spectrometer in order to meet the mass calibration criteria.

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

United States Environmental Protection Agency (EPA):
The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:
The process of substantiating specified performance criteria. (EPA-QAD)

Verification:
Confirmation by examination and provision of evidence that specified requirements have been met. (TNI)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Volatile Organic Compound (VOC):
An organic compound that is amenable to purge and trap analysis. In general, VOC have low boiling pints (<200^{o}C), high vapor pressures (tend to evaporate easily as low temperatures), and have low molecular weight (generally less than 300amu).

Work Cell:
A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Working Standard:
The standard that is analyzed on the instrument or using the analytical procedure. Also known as an intermediate reagent in TALS.

Acronyms:

ACRONYM	DEFINTION
A2LA	American Association for Laboratory Accreditation
AA	Atomic Absorption
AFCEE	Air Force Center for Environmental Excellence
AL	Action Level
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
bgs	Below Ground Surface
BNA	Base, Neutral, Acids (Semivolatile Organics)
BOD	Biochemical Oxygen Demand
BS	Blank Spike
BSD	Blank Spike Duplicate
BTEX	Benzene, Toluene, Ethylbenzene, Xylenes
BTU	British Thermal Unit
CA	Corrective Action
CAA	Clean Air Act
CAR	Corrective Action Report
CBOD	Carbonaceous Biochemical Oxygen Demand
CCB	Continuing Calibration Blank
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CDC	Continuing Demonstration of Capability
CDOC	Continuing Demonstration of Capability
CDQO	Chemical Data Quality Objective
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act

ACRONYM	DEFINTION
MRF	Method Request Form
MRL	Method Reporting Limit
MS	Mass Spectrometer
MS	Matrix Spike
MS/MS	Tandem Mass Spectrometry
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
MW	Monitoring Well
NBS	National Bureau of Standards
NCASI	National Counsel for Air and Stream Improvement, Inc.
NCM	Non-Conformance Module
NCR	Non-Conformance Report
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
nm	Nanometer
NPD	Nitrogen – Phosphorus Detector
NPDES	National Pollutant Discharge Elimination System
NPW	Non-Potable Water
ORO	Oil Range Organics
OSHA	Occupational Safety and Health Administration
OSTR	Outstanding SOP Training Report

ACRONYM	DEFINTION
CF	Calibration Factor
CFR	Code of Federal Regulations
CLLE	Continuous Liquid-Liquid Extraction
CLP	Contract Laboratory Program
COA	Certificate of Analysis
COC	Chain of Custody
COD	Chemical Oxygen Demand
CRDL	Contract Required Detection Limit
CRF	Change Request Form
CRQL	Contract Required Quantitation Limit
CSM	Corporate Safety Manual
CU	Custody
CVAA	Cold Vapor Atomic Absorption
CWA	Clean Water Act
DAI	Direct Aqueous Injection
DFTPP	Decafluorotriphenylphosphate
DM	Department Manager
DO	Dissolved Oxygen
DOC	Demonstration of Capability
DOD	Department of Defense
DOD QSM	Department of Defense Quality Systems Manual
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data Quality Objective
DRO	Diesel Range Organics
DU	Duplicate
DUP	Duplicate
DW	Drinking Water
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
EDQM	Environmental Data Quality Management
EHS	Environmental Health and Safety
EHSM	Environmental Health and Safety Manual

ACRONYM	DEFINTION
PAH	Polynuclear Aromatic Hydrocarbon
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
PCB	Polychlorinated Biphenyl
PDA	Photodiode Array
PDS	Post Digestion Spike
PE	Performance Evaluation
PGF	Peak Gaussian Factor
PID	Photoionization Detector
PM	Project Manager
PNA	Polynuclear Aromatic Hydrocarbon
PP	Project Plan
ppb	Parts Per Billion
PPE	Personnel Protective Equipment
PPL	Priority Pollutant List
ppm	Parts Per Million
ppq	Part Per Quadrillion
ppt	Parts Per Trillion
PQL	Practical Quantitation Limit
PRG	Preliminary Remediation Goals
PT	Proficiency Test
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl Chloride
PW	Potable Water
PWS	Public Water System
QA	Quality Assurance
QAM	Quality Assurance Manager
QAM	Quality Assurance Manual
QAMP	Quality Assurance Management Plan
QAN	Quality Assurance Navigator
QAP	Quality Assurance Plan
QAPjP	Quality Assurance Project Specific Plan
QAPP	Quality Assurance Project Plan
QAS	Quality Assurance Specialist

ACRONYM	DEFINTION
ELCD	Electrolytic Conductivity Detector
EPA	U.S. Environmental Protection Agency
ERPIMS	Environmental Resources Program Information Management System
eV	Electron Volt
FID	Flame Ionization Detector
FPD	Flame Photometric Detector
GALP	Good Automated Laboratory Practices
GC	Gas Chromatograph or Gas Chromatography
GC/MS	Gas Chromatograph/Mass Spectrometer
GE	General
GFAA	Graphite Furnace Atomic Absorption
GLP	Good Laboratory Practices
GPC	Gel Permeation Column (Gel Permeation Chromatography)
GRO	Gasoline Range Organics
HAA	Haloacetic Acids
HAPS	Hazardous Air Pollutants
HAZMAT	Hazardous Materials
HDPE	High Density Polyethylene
HECD	Electrolytic Conductivity Detector
HPLC	High Performance Liquid Chromatography
HRGC/HRMS	High Resolution Gas Chromatography/Hugh Resolution Mass Spectrometry
HT	Holding Time
HTRW	Hazardous, Toxic, and Radioactive Waste
HTV	Holding Time Violation
IC	Ion Chromatography
IC/EC	Ion Chromatography/Electric Conductivity
IC/MS	Ion Chromatography/Mass Spectrometer

ACRONYM	DEFINTION
QC	Quality Control
QCS	Quality Control Sample
QCSR	Quality Assurance Summary Report
QL	Quantitation Limit
QMP	Quality Management Plan
QSM	Quality Systems Manual
RCRA	Resource Conservation Recovery Act
RF	Response Factor
RI	Remedial Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
RRF	Relative Response Factor
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RT	Retention Time
RTW	Retention Time Window
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SD	Standard Deviation
SD	Sample Dilution
SD	Sample Duplicate
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SG	Semi Volatile Gas Chromatography
SIM	Selected Ion Monitoring
SM	Semi Volatile Mass Chromatography
SOC	Synthetic Organic Compound

ACRONYM	DEFINTION
ICAP	Inductively Coupled Argon Plasma Emission Spectroscopy
ICB	Initial Calibration Blank
ICCS	Interference Calibration Check Sample
ICOC	Internal Chain of Custody
ICP	Inductively Coupled Plasma
ICP/MS	ICP/Mass Spectrometer
ICS	Interference Check Sample
ICV	Initial Calibration Verification
IDC	Initial Demonstration of Capability
IDL	Instrument Detection Limit
IDOC	Initial Demonstration of Capability
IH	Industrial Hygiene
IPC	Instrument Performance Check Standard
IR	Infrared Radiation
IS	Internal Standard
ISO	International Standards Organization
ISTD	Internal Standard
LC	Liquid Chromatography
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LFB	Laboratory Fortified Blank
LFM	Laboratory Fortified Matrix
LFMD	Laboratory Fortified Matrix Duplicate
LIMS	Laboratory Information Management System
LM	Laboratory Manager
LOD	Limit of Detection
LOQ	Limit of Quantitation
LPC	Laboratory Performance Check
LQM	Laboratory Quality Manual
LRB	Laboratory Reagent Blank
LUFT	Leaking Underground Fuel Tank
LUST	Leaking Underground Storage Tank

ACRONYM	DEFINTION
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compound
SPE	Solid Phase Extraction
SPLP	Synthetic Precipitation Leaching Procedure
SR	Shipping and Receiving
SRM	Standard Reference Material
SS	Suspended Solids
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SVOC	Semi Volatile Organic Compound
SW-846	Solid Waste Analytical Protocols
TAL	Target Analyte List
TALS	TestAmerica LIMS System
TAT	Turn-Around-Time
TCL	Target Compound List
TCLP	Toxicity Characteristic Leachate Procedure
TDS	Total Dissolved Solids
TEPH	Total Extractable Petroleum Hydrocarbons
THM	Trihalomethanes
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TM	Technical Manager
TOC	Total Organic Carbon
TOX	Total Organic Halides
TPH	Total Petroleum Hydrocarbons
TRPH	Total Recoverable Petroleum Hydrocarbons
TS	Total Solids
TSD	Thermionic Specific Detector
TSS	Total Suspended Solids
TVPH	Total Volatile Petroleum Hydrocarbons
TVS	Total Volatile Solids
UCL	Upper Confidence Level

ACRONYM	DEFINTION
MB	Method Blank
MB	Microbiology
MBAS	Methylene Blue Active Substances
MCL	Maximum Contaminant Level
MCT	Maximum Conductivity Threshold
MD	Matrix Duplicate
MDL	Method Detection Limit
ME	Metals
µg/L	Microgram per Liter
mg/L	Milligram per Liter
MLG	Method Limit Group
µm	Micrometer
MPN	Most Probable Number

ACRONYM	DEFINTION
UCMR	Unregulated Contaminant Monitoring Rule
US EPA	United States Environmental Protection Agency
USACE	United States Army Corps of Engineers
USDA	United States Department of Agriculture
USGS	United States Geological Service
UST	Underground Storage Tank
UV	Ultraviolet
VG	Volatile Gas Chromatography
VM	Volatile Mass Chromatography
VOA	Volatile Organic Analysis / Volatile Organic Analyte
VOC	Volatile Organic Compound
ZHE	Zero Headspace Extraction

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Savannah performs work from clients located throughout the United States, as well as in some foreign countries. Most states and/or federal agencies maintain a laboratory accreditation program that requires a laboratory to obtain certification with their agency. To obtain certification, a laboratory must maintain an effective quality system that meets the requirements of the agency. Common components of the quality system requirements include maintaining up-to-date standard operating procedures (SOPs) and a Quality Assurance Manual (QAM); participating in a Proficiency Testing (PT) program; performing method detection limit (MDL) studies, initial and continuing demonstrations of capability studies (IDOCs/CDOCs), and internal assessments; and completing an annual renewal application. In addition to the requirements needed for certification, many agencies have specific analytical and/or reporting requirements that laboratories must follow.

Many agencies offer certification via reciprocity. Reciprocity is the acknowledgement of another state and/or agency's certification program. The most common types of reciprocity are homestate reciprocity and TNI (NELAC) reciprocity.

Lab Management, Project Management, Sales & Marketing, and the QA Manager may initiate requests for certification or accreditation. The QA staff completes the administrative tasks associated with the application and maintains the related documents in accordance with SOP SA-QA-001: *Document Control Program.*

Laboratory management has the responsibility and authority to ensure that laboratory operations are in compliance with program and regulatory requirements of the jurisdiction for which laboratory certification/accreditation is sought and maintained.

To perform compliance work in a particular state, the laboratory must maintain certification for the reported analytes. Most accrediting authorities will certify laboratories on a matrix/method/analyte level. For example:

Soil / EPA 8260B / benzene
Water / EPA 624 / toluene

Generally, laboratories must apply and submit supporting documentation (SOPs, MDLs, IDOCs, PTs, etc.) for each individual matrix/method/analyte combination.

1.0 Obtaining Certification

1.1 Certification Application Process

Lab Management, Project Management, Sales and Marketing, and the QA Manager may initiate requests for certification or accreditation. The application is obtained, reviewed, and completed by the QA Manager or designee. Sections of the application may be

distributed as appropriate to various staff members to assist in completion.

The certifying agency's regulations should be carefully reviewed at the time of application to ensure any non-routine requirements are communicated to the laboratory.

The QA Manager consults with Lab Management, Project Management, and the Sales and Marketing Staff to determine if additional methods should be added to the current laboratory certification in order to support existing and future work as the laboratory's capabilities change.

1.2 Reciprocity

Reciprocity is a means of acknowledging another agency's certification via mutual agreements between certifying agencies. Many certifying agencies offer some type of reciprocal certification. The most common types of reciprocity are based on either TNI (NELAC) certification or homestate certification.

Homestate reciprocity refers to another state's certifying agency allowing a laboratory to perform work in that state, provided that the laboratory maintains accreditation within the state in which it resides.

TNI refers to The NELAP Institute which governs the NELAP Standard document outlining Quality System and laboratory functions and requirements. NELAP refers to the National Environmental Laboratory Accreditation Program. Many states will acknowledge a laboratory's TNI accreditation from another state.

Note: Reciprocal agreements between states do not afford a "blanket" certification. To obtain reciprocal certification, a laboratory must still apply for accreditation, submit all required application materials, and receive notification of certification – usually in the form of a certificate - from the reciprocal agency.

1.3 Records Maintenance

A copy of the original application, certificate, and related materials are maintained in accordance with SA-QA-001: *Document Control Program.* Copies of current certifications are kept in the Certifications folder on the Public_QA drive, which is accessible to all laboratory staff. In addition, copies of current laboratory certifications from Savannah and other TestAmerica facilities are maintained in the TotalAccess marketing tool. These documents may be required to support subcontracting and marketing activities.

1.4 Maintaining Certification

Most states require continued evidence of an effective Quality System in order for a laboratory to maintain certification. In addition to annual renewal applications, laboratories are often required to complete bi-annual PT studies with acceptable results obtained for each certifiable matrix/method/analyte combination. Annual MDL and continued demonstrations of analyst capability are also routinely required, in addition to on-site assessments.

1.5 Certification Tools and Records

There are several tools in place to aid laboratory staff in determining what certifications the laboratory maintains and understanding any state-specific analytical and/or reporting requirements.

1.5.1 TotalAccess

Total Access is a tool that can aid in determining which certifications the laboratory maintains. This tool is useful in the pre-project planning process.

1.5.2 State and Project Requirement Summaries

Some states and/or projects have specific analytical and/or reporting requirements. A summary of these requirements is kept in the State and Project Requirement Summary on the Public-QA Drive. These requirements must be reviewed by project management and laboratory staff prior to initiating work. The Project Manager must clearly note in the TALS Worksheet Notes and/or Project Plan if the Project Requirement Summary (PRS) is to be followed.

1.6 Information Resources

1.6.1 Agency Information

The QA staff maintains a controlled access database that lists current contact information for the agency that oversees laboratory certification as well as the regulatory programs that are offered for certification by the agency. This information may be provided as a resource to Lab Management, Project Management, Corporate QA, and the Sales and Marketing staff.

1.6.2 Certification Matrix

The QA Department ensures that the certification matrix maintained on TotalAccess is current.

1.6.3 Certification / Accreditation Maintenance Requirements

Laboratory Management is responsible for ensuring that laboratory operations are in compliance with the regulatory and certification program requirements for the jurisdiction in which certification is maintained.

The QA Department is responsible for maintaining up to date applications and program information including program specific regulations and requirements.

Project Management is responsible for verifying certification of analytes and methods requested by the client prior to accepting work and should be familiar with the state-specific requirements of that state.

1.7 Certifications Listing

At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Authority	Certification Number or Laboratory ID Number
A2LA (DoD ELAP)	0399-01
A2LA (ISO/IEC 17025)	399.01
Alabama	41450
Alaska	UST-104
Arkansas	88-0692
California	3217CA
Colorado	N/A
Connecticut	PH-0161
Florida	E87052
Georgia	803
Georgia EPD	N/A
Guam	09-005r
Hawaii	N/A
Illinois	200022
Indiana	N/A
Iowa	353
Kentucky	90084
Kentucky UST	18
Louisiana	30690
Louisiana	LA160019
Maine	GA00006
Maryland	250
Massachusetts	M-GA006
Michigan	9925
Mississippi	N/A

Authority	Certification Number or Laboratory ID Number
Nebraska	TestAmerica-Savannah
New Jersey	GA769
New Mexico	N/A
New York	10842
North Carolina DENR	269
North Carolina PHL	13701
Oklahoma	9984
Pennsylvania	68-00474
Puerto Rico	GA00006
South Carolina	98001
Tennessee	TN02961
Texas	T104704185-08-TX
USDA	SAV 3-04
Virginia	302
Washington	C1794
West Virginia DEP	94
West Virginia DHHR (DW)	9950C
Wisconsin	999819810
Wyoming	8TMS-Q

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications. Copies of these documents can also be found on the laboratory's public server, in TotalAccess, and in the QA offices.

Revision History

Changes from the previous revision include:

- Updated Title Page and approved signatories.
- Updated Table of Contents.
- Updated SOP Listing.
- Updated references/requirements to incorporate SW846 Update V. Section 3
- Updated employee titles to reflect current designations. Section 4
- Included roles/responsibilities for Corporate ECO and Corporate QA employees. Section 4
- Updated laboratory employee responsibilities to be consistent with QAM template. Section 4
- Revised employee designated to serve as Laboratory Director Deputy to reflect QAM. Previously listed as CSM. Section 4.1
- Updated Corporate and laboratory Organization Charts. Section 4
- Updated laboratory's document control procedure to remove reference to QA Navigator. Replaced with reference to TALS File Shares.
- Added requirement that LD is responsible for reviewing and authorizing final acceptance of the quote for the facility. Section 7.1
- Removed requirement for Contracts Director and Proposal Coordinator to maintain copies of quote. Section 7.1
- Included reference to new Corporate Workshare SOP CA-C-S-001. Section 8
- Revised subcontract process to remove statement that client must provide acknowledgement that samples can be sent to another facility. Section 8
- Revised Workshare process to note that copy of the original COC is housed TALS, as opposed to requiring it to be shipped with the samples. Section 8.1
- Revised process for solvent and acid lot testing to denote that the approval information is located on the SharePoint directory on Oasis. Section 9.3
- Expanded responsibilities and process for receiving of materials and supplies. Incorporated requirements for verification of lots of acids and solvents. Section 9.2
- Removed QA Department as responsible party to maintain software verification. This responsibility lies with the IT Department. Section 9.3
- Expanded section on calibration of support equipment. Section 9.4
- Expanded section on and updated corporate SOP references for Data Recalls and Data investigations. Section 11
- Included reference to corporate Root Cause SOP. Section 12
- Added requirement that the QA Metrics Report is reviewed monthly by laboratory management, Corporate QA, and the EC. Section 13.1
- Expanded section on continuous improvement activities. Section 13
- Updated document control/archival process. Added note that QA records are maintained by the QA Department on the Q-Drive and are a part of the routine network backups. Removed requirement to document access to data with an access log. Removed reference regarding maintaining analytical data in hard copy. Removed reference to I-drive. Removed requirement to maintain run logbook per instrument. Section 14
- Removed reference to Target and MintMiner. Replaced with CHROM and AuditMiner. Section 15
- Expanded technical method audit process to incorporate data authenticity and analyst integrity audit such that each analyst is reviewed over the course of a 2 year period. Section 15
- Added requirement to review all SOPs annually. Section 15 and Section 19
- Added requirement to generate a written response for all PT failures. Section 19

- Revised MDL definition to denote as the value at which the analyst is 99% confident that the true value can be distinguished from blanks, as opposed to greater than zero. Section 19
- Expanded process/requirements for Login Review, First Level Review, and Second Level Review. Section 19
- Revised thermometer verification requirements to require bracketing temperature verifications only when thermometer range of use is greater than 10 degrees. Section 20
- Removed requirement to verify IR gun at a freezing temperature as this temperature is not encountered in normal use. Section 20
- Updated Instrument List. Section 20
- Removed reference to EPA Method 1653 as this method is no longer performed by the laboratory. Section 20 and Section 21
- Added requirement to verify mechanical pipettes daily for DOD. Added requirement to verify digital thermometers quarterly. Section 21
- Added requirements for verification of standards received without a Certificate of Analysis. Expanded process for receipt and documentation of reference standards. Section 21
- Removed text defining the first day of holding time as ending at 24 hours after sampling. Section 22
- Defined authorized test report signatories as the PM and PMA. Section 25
- Removed reference to faxing the final report. Section 25
- Added note that when NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Section 25
- Updated Laboratory Floor Plans.
- Updated location of laboratory certificates to reference Public_QA drive and TAACM. Removed reference to Public_G drive and Oasis. Updated Certifications Listing to reflect current certifications. Appendix 3

MICROEXTRACTABLES BY GC/ECD

(Methods: EPA 504.1 and 8011)

Approvals (Signature/Date):

Andrea Teal — May 27, 2015
Andrea Teal — Date
Quality Assurance Manager

Whitney Palefsky — April 23, 2015
Whitney Palefsky — Date
Environmental Health and Safety Coordinator

Kimberly Chamberlain — April 28, 2015
Kimberly Chamberlain — Date
Technical Manager

1.0 Scope and Application

This SOP gives the procedures for the determination of 1,2-Dibromoethane (Ethylene dibromide, EDB), 1,2-Dibromo-3-chloropropane (Dibromochloropropane, DBCP), and 1,2,3-Trichloropropane (1,2,3-TCP) in water samples by microextraction and gas chromatography/electron capture detection (GC/ECD).

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Thirty-five milliliters of sample are extracted with two milliliters of hexane. The extract is analyzed by gas chromatography utilizing dual capillary columns and dual electron capture (EC) detectors. Calibration standards are extracted and analyzed in the same manner as the samples.

This SOP is based on the following methods: EPA 504.1 and EPA 8011.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hexane and methanol must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences. Addition of sodium sulfate may be necessary to break up emulsions should they form during the extraction process.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Dibromochloromethane (DBCM) is a common disinfection byproduct in chlorinated drinking waters, often occurring in high concentrations. DBCM elutes closely to EDB, and, at high concentrations, may mask low concentrations of EDB. Adequate separation of DBCM and EDB must be demonstrated each day samples are analyzed.

4.2.4 High concentrations of non-target compounds may make detection and quantification of EDB, DBCP, and 1,2,3-TCP difficult. The electron capture detector is very sensitive to halogenated compounds and produces a very large response for concentrations as low as 1ppb. The results from the EPA 524.2 or EPA 8260 volatiles analysis can provide information about the compounds causing the interference. Some common volatile compounds that elute near the target compounds are tetrachloroethene, dibromochloromethane, chlorobenzene, bromoform, 1,2-dichlorobenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene. Note that very large concentrations of any halogenated solvent may overwhelm the response of the electron capture detector, making detection of the target compounds impossible at the routine reporting limit.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Hexane is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

Methanol is the primary solvent for standards. Hexane is used to extract the compounds from the samples. To minimize evaporation and the chance for exposure:

- store standards in methanol in glass containers with crimp-top caps and vials with Teflon-lined caps or septa
- store material with minimal headspace
- store materials at -10^{o}C or lower
- work under a hood
- if no hood is available, work in a well ventilated area, work quickly, and minimize the number of times the standard container is opened
- wear proper PPE (Personal Protection Equipment). PPE for this procedure includes a laboratory coat, eye protection, and gloves when handling standards, samples, or reagents.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Hexane	Flammable Irritant	500ppm TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

[1]Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Data System – Chemstation software is used to acquire and store data. CHROM software is used to reduce and output data. This software has the capability of processing stored GC data by recognizing a GC peak within any given retention time window and comparing the response of the peak to a reference standard. The software also allows calculation of response factors or construction of a calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

Gas Chromatograph - Agilent 6890 GC with 7683 autosampler, or equivalent, with dual micro electron capture detectors

Columns:

RTX CLP Pest, 30m x 0.32mm ID x 0.50um (Restek)
RTX CLP2, 30m x 0.32mm ID x 0.25um (Restek)
Guard Column Restek 5m x 0.32mm ID (Restek)

The columns are connected to a single injection port via a glass y-splitter and a 5m guard column. The ends of each column are connected to separate detectors. When properly configured, a single injection is split between the two columns to provide simultaneous detection and confirmation of the target compounds.

6.2 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 4 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Glass Transfer Pipettes	Various	Qualitative	Aliquotting sample extract	None	None
Mechanical Eppendorf-Style Pipettes (w/ Tips)	Various	QUANTITATIVE	Dilution Preparation	Monthly (Daily for DOD v5)	Accuracy = 2% Precision = 1%
Volumetric Flask (Class A)	Various	QUANTITATIVE	Calibration Standards Preparation	None (Class A)	None (Class A)
Gas Tight Autosampler Syringe	Various	QUANTITATIVE	Extract Injection; Standard and Spike Preparation	None, if received w/ COA	None, if received w/ COA
Autosampler Vial	2mL	Qualitative	Sample and Standard Containment	None	None
Sample Vial	12mL	Qualitative	Sample and Standard containment	None	None
VOA Vial	40mL 60mL	Qualitative	Standard and Spike containment	None	None
PTFE Squeeze Bottle	500mL	Qualitative	Dispensing Solvents	None	None
Pump-Style Mechanical Pipette	2mL	QUANTITATIVE	Dispensing Hexane	Monthly (Daily for DOD)	Accuracy = 2% Precision = 1%
Graduated Cylinder (Class A)	Various	QUANTITATIVE	QC Preparation	None (Class A)	None (Class A)
Erlenmeyer Flask	500mL	Qualitative	Extract Storage and Drying	None	None
Receiving Tip	10mL	Qualitative	Secondary Concentration Vessel	None	None

6.3 Lab Supplies

Residual (free) chlorine powder pack – HACH catalogue number 1539357 (for a 5mL sample)

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

40mL VOA vial, with sodium thiosulfate dechlorination agent – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041*: Reagent and Standard Materials Procedures.*

Hexane and methanol must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Purchased Reagents

7.2.1.1 Laboratory Reagent Water – ASTM Type II

Note: If laboratory water is unsuitable for analysis of target compounds, purging the water with nitrogen for 30 minutes or boiling the water and purging with nitrogen may reduce contamination.

7.2.1.2 Hexane – residue grade
TALS Name: Hexane(lot number)_
Storage: Room temperature
Expiration:
Unopened: 5 years or manufacturer's expiration date
Opened: 6 months

7.2.1.3 Methanol – residue grade
TALS Name: EX_MEOH
Storage: Room temperature
Expiration:
Unopened: 5 years or manufacturer's expiration date
Opened: 6 months

7.1.4 Sodium Chloride (NaCl) - ACS reagent grade. Purify by heating at 400°C for four hours in a shallow tray.
TALS Name: NACL
Storage: Room temperature
Expiration:
Unopened: 5 years or manufacturer's expiration date

Opened: 5 years or manufacturer's expiration date; 6 months from baking.

7.1.5 Sodium Sulfate - granular, anhydrous - Purify by heating at 400°C for four hours in a shallow tray.
TALS Name: EX_na2SO4
Storage: Room temperature
Expiration:
Unopened: 5 years or manufacturer's expiration date
Opened: 5 years or manufacturer's expiration date; 6 months from baking.

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and attached to TALS.

The standard recipes are included in Attachment 5. This attachment includes stock standards (vendor and part number) used in this SOP, the preparation steps for the intermediate and working standards, and the instructions for assigning expiration dates to the stocks, intermediate, and working level standards.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Samples are routinely collected in 40mL VOA vials containing 75uL of a 40mg/mL solution of sodium thiosulfate de-chlorination agent. The dechlorination agent should be sufficient to remove residual chlorine from the sample. Samples should be collected without headspace.

Note: 40mL VOA vials with HCl preservative may also be used for EPA 8011. This bottle type is consistent with the bottle type used commonly used for analysis of these particular analytes by EPA 8260.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored at in the refrigerator at 0-6°C (less than 6°C but not frozen) until the time of analysis and analyzed within 24 hours of extraction.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives and/or de-chlorination agents. NCMs must be initiated for samples that are received containing headspace.

8.1.1 Preservation Checks – Residual Chlorine

These checks are performed prior to preparation.

8.1.1.1 Mix the sample by inverting and transfer 6mL to a small medicine cup.

8.1.1.2 Add a residual chlorine powder pillow to the sample in the cup and note the presence of a pink color, which indicates the presence of residual chlorine.

If the sample tests positive for residual chlorine, initiate an NCM noting that residual chlorine was present.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An extraction batch consists of up to 20 environmental samples and the associated QC items extracted together within a 24 hour period.

9.1.1 For EPA 504.1 and EPA 8011, the laboratory's default minimum QC items performed for each extraction batch are: a method blank, laboratory control sample (LCS), a low-level LCS (LLCS) at the method detection limit (MDL), a matrix spike (MS) per 10 samples, and a matrix spike duplicate (MSD).

Note: This procedure incorporates extracted calibration standards; therefore, initial and continuing calibration standards are also prepared within the extraction batch. Refer to Attachment 5 for standard preparation instructions.

9.1.2 The routine container supplied for this method is a 40mL container. 35mL is required for extraction. Due to the nature of this procedure, and the need to maintain zero headspace, reduced sample initial volumes must not be used to achieve the required batch matrix spike frequency (i.e., a separate vial is required for to perform each of the native sample, MS, and MSD analyses). As such, an MS/MSD can only be prepared when additional containers are provided.

9.1.3 If there is insufficient sample volume to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of three 40mL VOA vials for matrix spike/matrix spike duplicate and less than two 40mL VOA vials for the additional matrix spike required for batches having greater than 10 samples.

9.1.4 Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Instrument QC

9.2.1 Column Resolution / RL Check

The purpose of the Column Resolution / RL Check is check is to ensure that EDB can be resolved from a common chlorination by-product, dibromochloromethane (DBCM). DBCM is often present at concentrations that are much higher than EDB is expected to occur, and this check is intended to demonstrate that EDB can be detected at the RL of 0.020ug/L in the presence of DBCM at 1.0ug/L, a 50-fold difference in concentrations.

- Prepare, extract, and analyze the Column Resolution Check standard (Table 5).

- Evaluate the chromatogram, inspecting the resolution between DBCM and EDB on both columns.

- The peaks should be resolved at the baseline (a gap in the baseline from the end of the DBCM peak to the beginning of the EDB peak) on both columns.

- If EDB cannot be detected, prepare a new standard and repeat the preparation and analysis. If the repeat analysis still does not meet the criterion, take steps to increase the resolution between these two compounds which may include decreasing the initial temperature and/or reducing the column flow rate.

Note: Do not proceed with the analysis is this check can be met.

9.2.2 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 5 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted.

The initial calibration standard concentrations currently in use in the laboratory are listed in Attachment 5. Refer to Attachment 5 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

Note: This procedure incorporates extracted standards; therefore, initial and continuing calibration standards are prepared within the extraction batch. Once the ICAL is established, subsequent CCVs must be extracted each day samples are extracted. All ICAL, ICV, and CCV standards must be analyzed within 24 hours of extraction.

9.2.2.1 ICAL Criteria

The preferred method of quantitation is the average response factor. The relative standard deviation (%RSD) of the calibration standards must be <10% for EPA 8011 and <20% for EPA 504.1 for the initial calibration curve to be acceptable.

If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. If the regression curve option is chosen, the regression coefficient (r^2) must be greater than or equal to 0.990 to be acceptable.

If these criteria are not met, then re-calibration is required before sample analysis can proceed.

9.2.3 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified after the initial calibration is established, prior to any sample analyses, in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to Level 5 of the ICAL. Refer to Attachment 5 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

The ICV must be within 30% of the true value to be acceptable.

Note: If the LCS is prepared from a second source standard it can be used to satisfy the ICV criteria.

9.2.4 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The method blank for this method is analyzed and evaluated in lieu of instrument or calibration blanks.

Additional instrument blanks may be analyzed after samples with high levels of target or non-target compounds to mitigate and evaluate the analytical system for carry-over.

9.2.5 Continuing Calibration Verification

The initial calibration curve must be verified at the beginning and end of every 12-hour clock for EPA 504.1 and at the before and after every 20 samples analyzed for EPA 8011.

The concentration of the standard should be varied, such that several points of the calibration range are verified.

The CCV must be within 30%D to be acceptable for EPA 504.1.

The CCV must be within 20%D to be acceptable for EPA 8011.

9.2.6 Surrogate

This procedure uses a surrogate compound to evaluate the extraction process. Pentachloroethane is the surrogate used for this procedure. Another surrogate compound may be used provided it produces consistent results within method-defined criteria.

Prior to preparation, this surrogate is added to all samples and QC items. The concentration of the surrogate is the same in all field samples and QC samples. A concentration of 0.50ug/L is used.

The percent recovery of the surrogate in all field samples and QC samples must be within the limits listed in the Method Limit Groups (MLGs) in TALS. If the percent recovery is

outside of this range, the analysis of the sample must be repeated. Repeated failure of the surrogate percent recovery may indicate re-extraction is necessary.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

Samples, calibration standards, and QC items are subjected to the same extraction and analytical procedures.

10.1.1 Remove the samples from storage and scan them into the SG Department. Allow them to warm to room temperature.

10.1.2 Gather and label one 40mL VOA vial for each calibration standard and QC item. Add 35mL reagent water to each of the labeled VOA vials. Prepare the calibration standards in accordance with Attachment 5. Prepare the QC items in accordance with Section 10.2.

10.1.3 Scan each sample into the prep batch and complete the information for the calibration standards and the QC items for the batch.

10.1.4 Inspect the samples for large air bubbles, the presence of large amounts of sediment, and other anomalies, and, if present, contact the Department Manager to determine the course of action.

In the absence of any additional guidance, use the following:

- If the sample contains air bubbles, notify the Project Manager via a Nonconformance Memo (NCM) and proceed with the analysis.
- If the sample contains large amounts of sediment, pour off the liquid above the sediment into a tared 40mL vial and proceed with the analysis. Notify the Project Manager via an NCM.

10.1.5 Working with each sample in turn, invert the sample vial three times and open the cap. Pipette 6mL of sample from each 40mL sample vial and transfer to a labeled, 25mL plastic cup. Cap the vial. Repeat for the remaining samples.

Add a residual chlorine powder pack to the 6mL of sample that was transferred to the plastic cup. Note whether the residual chlorine test is positive (pink color forms) or negative (no color forms) on the prep sheet. If residual chlorine is present, an NCM must be initiated.

10.1.6 Weigh and record the weight of the capped sample vials (after removing 6mL) to the nearest 0.1 grams on the prep sheet.

10.1.7 Add the surrogate spiking solution to each sample and QC item as follows:

- draw 35uL of the surrogate spiking solution into a 50uL syringe
- inject the surrogate spiking solution, through the vial septum, under the surface of the sample
- invert the sample once to mix
- repeat for the remaining samples and QC items.

10.1.8 Add the 504 Spike Solution to each LCS/LCSD and MS/MSD item as follows:

- draw 35uL of the 504 Spike Solution into a 50uL syringe
- inject the 504 Spike Solution, through the vial septum, under the surface of the sample
- invert the sample once to mix
- repeat for the remaining QC items.

Add the WS#1 Spike Solution to the LLCS as follows:

- draw 10uL of the WS#1 Spike Solution into a 10uL syringe
- inject the WS#1 Spike Solution, through the vial septum, under the surface of the sample
- invert the sample once to mix
- repeat for the remaining QC items.

10.1.9 Working with one vial at a time, quickly remove the cap, add 6g of purified sodium chloride (NaCl), then pipette 2.0mL hexane into the vial for each standard, sample, and QC item. Recap the sample containers and gently swirl until the NaCl has dissolved.

10.1.10 Shake the vials for approximately three minutes by hand, or for approximately 5 minutes using a shaker table set on high.

10.1.11 Allow the hexane and water layers to separate.

10.1.12 Remove the cap and carefully transfer approximately 400uL of the extract (i.e., hexane; top layer) into a GC autosampler vial fitted with a 400uL insert. Cap the vial. The extract is now ready for analysis.

Note: If an emulsion forms in the solvent layer, add small amounts (~0.1g) of purified sodium sulfate to the extract, letting the crystals fall gently through the emulsion/solvent layer.

If the sodium sulfate does not break the emulsion, centrifuge the sample for approximately three minutes. The centrifuge is located in the 508 Prep Laboratory. If centrifugation does not clear up the emulsion, freeze the sample to separate the water layer and solvent layer.

Invert the remaining sample and vials and store in the refrigerator at 0-6^{o}C until analysis is completed. The extracts must be analyzed within 24 hours of extraction.

10.1.13 Determine the volume of sample as follows:

- remove the cap and pour the sample into a separatory funnel to separate the hexane from the water. Discard the water sample (bottom layer) down the sink then pour the remaining hexane (top layer) into the flammable waste container.
- "flick" the sample vial several time to remove the remaining drops of water
- recap the vial, weigh the vial, and record the weight of the empty sample container on the prep sheet to the nearest 0.1g.

10.1.14 Calculate the volume of sample, assuming that 1.0g of sample is equal to 1.0mL of sample.

$$V = (W_1 - W_2) \otimes \frac{1.0mL}{g}$$

Where:
V = volume of sample extracted (mL)
W_1 = weight of vial, cap, and sample (g) (Section 9.6)
W_2 =weight of empty vial and cap (Section 9.13)

Record the volume of sample on the prep sheet.

10.1.15 Complete the TALS prep sheet.

10.2 QC Sample Preparation

Refer to Section 9.1 for the minimum QC items to be prepared with each preparation batch of twenty or fewer field samples. Additionally, the Resolution Check / RL Standard and a mid-level CCV (or multi-point ICAL) are also required.

Note: The LCS/LCSD are prepared using a source different from the source used to the calibration standards and also serve the initial calibration verification standards for an ICAL.

Batch QC samples are processed in the same manner as field samples.

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

Two dissimilar columns are connected to the injection port using a press-tight glass y-splitter and a guard column which provides simultaneous detection and confirmation of the target analytes.

These conditions and parameters are given for guidance and for a starting point if the method is lost in the acquisition computer. The conditions and parameters may be

modified to optimize the analytical system. The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the MDL.

GC Parameters
Column1: Restek CLPesticides 30m x 320um, x 0.50um
Column2: Restek CLPesticides 2 30m x 320um, x 0.25um
Injector: 240°C
Mode: Pulsed Splitless
Pressure: 25.14psi
Pulse pressure: 50.0psi
Pulse time: 0.30 minutes
Purge flow: 55.9mL/minute
Purge time: 0.28 minutes
Total flow: 94.9mL/min (hydrogen)

Temperature program
Initial Temp: 55°C
Initial Hold: 1.60 minute
Program Rate: 76.33°C per minute to 95°C, hold 1.00 minutes
60.00°C per minute to 150°C hold 1.29 minutes
Maximum Temp: 320°C

Run Time: Approximately 5.33 minutes

Detector: Dual electron capture
Detector temperature: 305°C
Makeup flow: 80mL/min (nitrogen)

Signal data rate: 10hz

Autoinjector
Sample washes: 0
Sample pumps: 4
Injection volume: 2.0uL
Syringe size: 10uL
PostInj(ection) Solvent A washes: 6
PostInj(ection) Solvent B washes: 6 (use hexane as wash solvent)
Viscosity delay: 0 seconds
Plunger speed: fast
Preinjection dwell: 0.00minutes
Postinjection dwell: 0.00minutes

10.3.1.1 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP SA-QA-008: *Evaluation of Chromatographic Data*. Retention time windows (RTW), i.e., the length of time the instrument will scan for the analyte, must be established initially upon instrument set-up and verified annually.

Retention times (RT), i.e., the elution time of the analyte, are verified daily with the analysis of the ICAL or CCV. The retention time for the CCV must fall within the daily retention time window as defined in SOP SA-QA-008.

10.3.2 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.2. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.3, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5.

10.3.3 Sample Analysis

Remove the extracts from the refrigerator and allow them to come to room temperature.

The sample extract must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The default procedure is to exclude QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in the clock.

10.3.4 Example Analytical Sequence

Refer to Attachment 1 for an example analytical sequence.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data.* This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

11.1.1 Target Analyte Identification

The judgment and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP SA-QA-008: *Evaluation of Chromatographic Data* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

11.1.2 Manual Integrations

Manual integrations must be documented in accordance with SOP SA-QA-008. Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

11.1.3 Dual Column Reporting

Refer to SOP SA-QA-008: *Evaluation of Chromatographic Data* for information on assessing and reporting data from dual columns.

11.1.4 Surrogate Evaluation

The surrogate, pentachloroethane, is spiked into each sample and QC item prior to preparation. Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Evaluate the surrogates in the same manner as the target compounds using the guidance above.

Refer to Section 11.1.5.1 for information on the surrogate dilution threshold factor.

Note: Other surrogate compounds may be used provided they produce consistent results within method-defined criteria.

11.1.5 Dilutions

If the response for an analyte exceeds the working range of the system, a dilution is required. Prepare dilutions of the extract if the dilution can be analyzed within 24 hours of the time the sample was extracted. If not, extract a smaller aliquot of sample and repeat the analysis.

Dilution from Extract

Dilution Factor	Volume of Extract	Final Volume of Dilution in Hexane
2	500uL	1000uL
5	200uL	1000uL
10	100uL	1000uL
25	40uL	1000uL
50	20uL	1000uL
100	10uL	1000uL

Dilution from Sample

Dilution Factor	Volume of Sample	Final Volume of Dilution in Water
2	25mL	50mL
5	10mL	50mL
10	5.0mL	50mL
25	2.0mL	50mL
50	1.0mL	50mL
100	0.5mL	50mL

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

11.1.5.1 Surrogate Dilution Threshold Factor

Surrogates may be diluted out if the concentration of target compounds is high or the presence of non-target compounds interferes with the quantification of the target compounds. Undetect surrogates in the sample when the dilution factor is 6 or greater. As such, recoveries must be reported as “0D”, and control limits will not apply.

An NCM must be initiated to denote this situation.

11.1.5.2 Dilutions and MS/MSD Recoveries

Matrix spike recoveries are not reported for dilutions of 6 or greater. An NCM is generated for instances where the dilution prohibits evaluation of the MS/MSD recoveries. In instances where the unspiked sample concentration is more than four times the concentration of the target compound spiked into the MS and MSD, the results are qualified with “4” or other suitable flag.

An NCM must be initiated to denote this situation.

11.1.6 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes or use the TALS Historical Date tracker feature to determine if historical data is available for review.

11.1.7 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCLs associated with this procedure are given in Attachment 6. Notify the PM immediately via a Nonconformance Memo if any sample contains a detection above these levels.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample (at the instrument)
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor

Note: This calculation assumes all applicable unit correction factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: EPA 8011specifies that the MDL must be ≤0.03ug/L.

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS) for EPA 504.1 are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits for EPA 504.1 and does not utilize in-house nor laboratory-derived limits for the evaluation of batch QC items.

The control limits for the batch QC items (LCS, MS) for EPA 8011 are not specified in the reference method; therefore, the laboratory utilizes in-house nor laboratory-derived limits for the evaluation of batch QC items for EPA 8011.

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017.

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four

consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 13 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Flammable waste (acetone, hexane, and methanol from extracts, rinsings, and standards) - Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.
- Sample residue from the sample vials contains hexane - The samples are poured into a separatory funnel that is used to separate the hexane layer from the aqueous layer. The aqueous layer is discarded down the sink, and the hexane layer is contained in a flammable waste container.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, December 1986 and February 2007.
  - Method 8011: 1,2-Dibromoethane and 1.2-Dibromo-3-Chloropropane by Microextraction and Gas Chromatography, Revision 0; July 1992
  - Method 8000B: Determinative Chromatographic Separations, Revision 2; December 1996.
- *Methods for the Determination of Organic Compounds in Drinking Water:*
  - EPA Method 504.1, 1,2-Dibromoethane (EDB), 1.2-Dibromo-3-Chloropropane (DBCP), and 1,2,3-Trichloropropan (123TCP) in Water by Microextraction and Gas Chromatography, Revision 1.1, Munch, J.W. 1995

16.0 Method Modifications and Clarifications

16.1 The reference method was written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure.

16.2 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this frequency via the Low-level LCS required by both methods.

16.3 The amount of sodium chloride added to the samples differs between EPA 504.1 (6g) and EPA 8011 (7g). This SOP directs the analyst to use 6g per sample, calibration standard, and QC item. The addition of salt to the sample is to increase the polarity of the sample matrix, which increases the tendency for the target compounds to partition into the non-polar solvent (hexane). Six grams of salt is adequate to achieve this purpose. In addition, the laboratory believes this minor modification of the method to have no impact on sample results since the samples and calibration standards are processed in the same manner.

16.4 A calibration standard at one half the routine RL of 0.020ug/L is included in the initial calibration to support the RL of 0.010ug/L required by one or more state agencies.

16.5 The laboratory has incorporated the batch QC items as outlined in Section 9.1. Some additional QC items are performed (above those required in the reference methods) to

satisfy common state regulatory and/or client requests for precision data and/or to facilitate scheduling and data evaluation. Additionally, some QC items are combined (such as the daily Low-level LCS required for the EPA Drinking Water Manual and the weekly Low-level LCS required by EPA 504.1; or for EPA 504.1, the CCV and the LCSD required for batches of 11-20) to facilitate analysis and performing both EPA 504.1 and EPA 8011 in the same batch.

The method-specified batch QC items are as follows:

EPA 504.1: Lab reagent blank and field reagent blank each day; 1 LCS per 10% of samples (70-130%R); Low-level LCS weekly (60-140%R); 1 MS per batch (65-135%).

EPA 8011: reagent and calibration blank per batch; check sample at 0.25ug/L for 5% of samples (60-140%R); QC reference sample at 0.10ug/L weekly (60-140%R); MS/MSD or sample duplicate daily.

EPA Manual for the Certification of Laboratories Analyzing Drinking Water: method blank per batch, LCS per batch, Low-Level LCS daily.

16.6 EPA 8011 does not give CCV acceptance criteria. The laboratory uses 20%D, which is consistent with the guidance given in EPA 8000C.

16.7 EPA 504.1 includes the use of Field Reagent Blanks (i.e., trip blanks). The laboratory does not normally include these in outgoing bottle kits; however, this task can be accommodated upon client request.

16.8 SW-846 does not specifically address bottle types for EPA 8011. The bottle type specified in EPA 504.1 (i.e., a 40mL VOA vial with sodium thiosulfate dechlorination agent) can be used for both EPA 504.1 and EPA 8011, or alternatively, the bottle type specified in EPA 8260 (i.e., a 40mL VOA vial with HCl preservative) or that specified in EPA 624 (i.e., a 40mL unpreserved VOA vial) can be used for this method.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Standard Preparation Recipes
Attachment 6: Maximum Contaminant Level (MCL) Table
Attachment 7: Labware Cleaning Procedures

Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary
Thirty-five milliliters of sample are extracted with two milliliters of hexane. The extract is analyzed by gas chromatography utilizing dual capillary columns and dual electron capture (EC) detectors. Calibration standards are extracted and analyzed in the same manner as the samples.

Example Analytical Sequences
Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Instrument Blank	Hexane
ICAL	Minimum of five points
Instrument Blank	Hexane
ICV	Second source standard
Instrument Blank	Hexane
Field and QC samples	Not to exceed 20 field samples
CCV	Mid-level
Instrument Blank	Hexane
Field and QC	Not to exceed 20 field samples
CCV	Mid-level calibration standard
Instrument Blank	Hexane

Analytical Sequence for samples not immediately following an initial calibration:

Description	Comments
Instrument Blank	Hexane
RL Standard	Per batch
CCV	Mid-level calibration standard
Instrument Blank	Hexane
Field and QC Samples	Not to exceed 20 field samples
CCV	Mid-level
Instrument Blank	Hexane
Field and QC Samples	Not to exceed 20 field samples
CCV	Mid-level
Instrument Blank	Hexane

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Listed below are the holding times and preservation requirements:

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Water	3 x 40mL VOA; no headspace	35mL	35mL	None	0-6°C	Sodium Thiosulfate	Extraction: 14 days from collection Analysis: 24 hours from extraction

Attachment 3: QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) - Minimum 5 points -Extracted	Analyzed initially prior to sample analysis, when major instrument maintenance performed, or when CCV fails	EPA 504.1: %RSD<20% r^2>0.990 EPA 8011: %RSD<10% r^2>0.990	Refer to SOP SA-QA-016
Initial Calibration Verification (ICV) - 2nd Source - Extracted	Analyzed after each ICAL. Note: LCS is used to satisfy since from a second source.	70-130% Recovery	Refer to SOP SA-QA-016
Continuing Calibration Verification (CCV)	Extracted each day samples are prepared. Analyzed initially, after every 20 samples (not to exceed 12 hours), and at the end of the sequence - Concentration must be varied throughout the mid-range.	EPA 504.1: <30%D EPA 8011: <20%D	Refer to SOP SA-QA-016
Calibration Blank (CCB/ICB)	After ICV and every CCV	<MDL	Terminate the analysis; correct problem; reanalyze affected samples.
Surrogate	All field, batch QC, & instrument QC samples	Within TALS MLG limits	Refer to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	Extracted together w/in 24-hr period; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	<MDL	Refer to SOP SA-QA-017
Laboratory Control Sample (LCS)	One per batch	Within limits listed in the MLG	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient volume provided for MS/MSD	Within limits listed in the MLG	Refer to SOP SA-QA-017
Low-Level Laboratory Control Sample (LLCS) / Method Detection Limit Verification (MDLV)	One per batch	60-140%R	Refer to SOP SA-QA-017
Matrix Spike (MS)	One per batch	Within limits listed in the MLG	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	One per batch	Within limits listed in the MLG	Refer to SOP SA-QA-017
Column Resolution Check (DBCM Check)	One per batch	Baseline resolution between DBCM and EDB	- Perform column maintenance - Adjust column flow/temperature to attain resolution. - Install new column or columns.

QC Item	Frequency	Criteria	Corrective Action
Retention Time Window Determination	Annually	Refer to SOP SA-QA-016	Refer to SOP SA-QA-016
Initial Demonstration of Capability (IDOC)	Initially; Per analyst / matrix / method / analyte combination	Within limits listed in the MLG	Refer to SOP SA-QA-006 (Unsupervised work must not begin without successful completion of IDOC.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Within limits listed in the MLG	Refer to SOP SA-QA-006
Method Detection Limit (MDL)	Upon method/instrument set-up	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, and quarterly thereafter	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

Refer to the instrument manufacturer's guides for trouble-shooting items.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Guard Column/Injector							X	Change sleeve and cut front of guard column, recommended daily
Septum							X	Replace, recommended daily
Splitless Disc							X	Replace, recommended daily
Autosampler							X	Syringe cleaned or replaced as needed
Column							X	Change column

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Attachment 5:
Standard Preparation Recipes

Stock Standard Mixes

Stock/Mix	TALS ID	Vendor/ Part Number	Concentration (ug/mL)
504.1 Mixture	SG504ICV_	Ultra DWM-514	200
552.2 Internal Standard (1,2,3-Trichloropropane Soln.)	SG123TCP_	Ultra PPS251-1	1000
504.1 EDB/DBCP Spike Std (Second Source)	SG504CAL_	Accustandard M-504	200
Dibromochloromethane (Stock)	SG504DBCM_	Ultra HC-100	100
504 Surrogate (Pentachloroethane)	SGPCE504_	Restek 30404	2000

Storage: <-10℃
Expiration:
Unopened: Manufacturer's expiration date
Opened: 6 months from opening

504 Intermediate Standard (TALS ID = 504 INT A)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
504.1 Mixture (SG504CAL_)	125	2.0	12.5 (EDB/DBCP)
552.2 Internal Standard (SG123TCP_)	125		62.5 (1,2,3-TCP)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504 Working Standard #1 (TALS ID = 504 WS#1)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
504 Intermediate A (504 INT A)	50	10	0.0625 (EDB/DBCP) 0.3125 (1,2,3-TCP)
504 Surrogate (504 Penta_)	500		0.025 (PCE)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504 ICV/LCS Spike Intermediate Standard (TALS ID = 504 Spike_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
504.1 EDB/DBCP Spike Std (Second Source) (SG504ICV_)	12.5	25	0.10 (EDB/DBCP)
552.2 IS (SG123TCP_)	10		0.50(123TCP)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504 Surrogate Intermediate (TALS ID: 504 Penta_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
SGPCE504_	25	100	0.50 (PCE)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504 Pentachloroethane Spiking Solution (TALS ID: 504_Surr_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
504 Penta_	500	10	0.025 (PCE)

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

Dibromochloromethane Intermediate Standard (TALS ID = 504-DBCM_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)*	Final Concentration (ug/mL)
Dibromochloromethane (Stock) SG504DBCM	5.6	2	0.28

(*) in methanol
Storage: <-10℃
Expiration: 1 month from prep date

504/8011 Calibration Standards (TALS ID: enter in prep batch)

CAL STD	Volume 504 DBCM (uL)	Volume 504 WS#1 (uL)	Volume of Reagent Water (mL)	EDB/DBCP		1,2,3-TCP		DBCM
				ug/mL[1]	ug/L[2]	ug/mL[1]	ug/L[2]	ug/mL[1]
1	5	5	35	0.000156	0.0089	0.000781	0.045	0.0175
2	0	10	35	0.000313	0.018	0.001563	0.089	0
3	0	20	35	0.000625	0.036	0.003125	0.18	0
4	0	35	35	0.001094	0.063	0.005469	0.31	0
5	0	50	35	0.001563	0.089	0.007813	0.45	0
6	0	65	35	0.002031	0.12	0.010156	0.58	0
7	0	80	35	0.0025	0.14	0.0125	0.71	0
8	0	100	35	0.0031250	0.18	0.015625	0.89	0

Storage: Not applicable; made fresh each day
Expiration: 24 hours

(1) Concentration in extract, final volume = 2.0mL
(2) Concentration in standard, initial volume = 35mL

504/8011 Initial Calibration Verification/LCS (TALS ID: enter in prep batch)*

STD	Volume 504 Spike (uL)	Volume 504 Surr DL (uL)	Volume of Reagent Water (mL)	Final Concentration (EDB/DBCP) (ug/L)	Final Concentration (1,2,3-TCP) (ug/L)
ICV/LCS	35	35	35	0.10	0.50

Storage: Not applicable; made fresh each day
Expiration: 24 hours
*Also used to prepare the MS and MSD

504/8011 Column Resolution/RL Check (TALS ID: enter in prep batch)

STD	Volume of Intermediate (uL)		Volume of Water	Concentration of RL/Resolution Check (ug/L)		
	504 INT A	504-DBCM		EDB/DBCP	1,2,3-TCP	DBCM
RL/ResCheck	5	5	35mL	0.010	0.050	1.0

Storage: Not applicable; made fresh each day
Expiration: 24 hours

Guidance for Preparing Intermediate and Working Standards in Methanol

- Clean and rinse volumetric flask with methanol.
- Add methanol to volumetric flask to approximately one half volume.
- Add standard to volumetric flask, inserting the syringe needle under the surface of the methanol.
- Dilute to volume with methanol, cap, and invert three times to mix.
- Transfer by gently pouring the newly made standard into a labeled storage with minimal headspace and seal with Teflon-lined screw or crimp cap.
- Store at -10C in freezer

Guidance for Preparing Calibration and Verification Standards in Water

- Add 35mL of reagent water to a 40mL VOA vial.
- Add standard to the vial, inserting the syringe needle under the surface of the water.
- Cap the vial and mix by inverting three times.
- Use immediately.

Attachment 6:
Maximum Contaminant Level (MCL) Table

Primary Drinking Water Regulations		
Contaminant	**MCL (mg/L)**	**MCL (ug/L)**
1,2-Dibromo-3-chloropropane (DBCP)	0.0002	0.2
Ethylene Dibromide (EDB)	0.00005	0.05

Attachment 7: Labware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES

SEMIVOLATILE GC LAB

1. Scrub with tap water and Liquinox.
2. Rinse 3 times thoroughly with tap water.
3. Rinse 3 times thoroughly with Acetone.
4. Place volumetric top-down within storage rack and allow to air dry.
5. Store in closed drawer.

FSG046:12.04.13:0

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18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Added Volumetric Container Table. Section 6.2
- Adjusted sample collection and storage conditions to reflect 0-6°C. Section 8.1
- Removed requirement to extract an ICAL and ICV with each batch of samples. Once the ICAL is established, subsequent CCVs must be extracted each day samples are extracted. Section 9.2.2 and Attachment 3
- Changed CCV criteria for EPA 8011 from 15%D to 20%D. Section 9.2.5, Section 16.6, and Attachment 3
- Revised standard preparation instructions, concentrations, and nomenclature to reflect current laboratory practice. Attachment 5
- Added Labware Cleaning Procedures. Attachment 7

VOLATILE COMPOUNDS IN DRINKING WATER BY GC/MS

(Methods: EPA 524.2)

Approvals (Signature/Date):

Andrea Teal 12/04/2015
Andrea Teal Date
Quality Assurance Manager

Whitney Palefsky 12/04/2015
Whitney Palefsky Date
Environmental Health & Safety Coordinator

Charlton A. Riegner 12/04/2015
Charlton Riegner Date
Technical Manager

1.0 Scope and Application

This SOP gives the procedures for the determination of volatile organic compounds in water samples by gas chromatography/mass spectrometry (GC/MS).

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOC onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

This SOP is based on the following method: EPA 524.2.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

THM (Trihalomethanes) - The four THM are chloroform, dichlorobromomethane, dibromochloromethane, and bromoform.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions (Attachment 8) to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Methanol must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 VOC commonly used in the laboratory may be a major source of contamination. Hexane, methylene chloride, acetone, freon, 2-butanone (MEK), toluene, and isopropanol are all common laboratory solvents and tend to cause the most interference. The analyses of highly concentrated samples (>1ppm) may also affect the succeeding runs. “Carryover” can occur when low concentration samples are analyzed after high level samples. Reagent blanks must be analyzed periodically to check for laboratory contamination and carryover. The VOC laboratory must be kept as free from contaminants as possible.

4.2.4 Samples containing chlorine must be treated with ascorbic acid. If excess chlorine is not destroyed, the concentration of some compounds formed when water is chlorinated (for example, trihalomethanes) may not reflect the analyte concentration at the time of sampling. Samples for trihalomethanes are dechlorinated using sodium thiosulfate.

4.2.5 Samples must be acidified, except samples where only trihalomethanes are requested, at the time of collection (after dechlorination) to prevent biological degradation of some VOC. The addition of acid also minimizes dehydrohalogenation of some chlorinated alkanes.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye

protection (safety glasses or face shield), disposable nitrile gloves (or equivalent), and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Hydrochloric acid is extremely hazardous as an oxidizer, a corrosive, and a poison, and it is reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and may lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage. Concentrated acids should be used in a fully functional fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP.

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the “MSDS” link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hydrochloric Acid	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Analytical Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Agilent (HP) 5973 Mass spectrometer equipped with a capillary direct interface.

Agilent (HP) 6890 Gas chromatograph with spilt/splitless injector. The exit vent must have a carbon trap in-line to collect the volatile compounds that are vented during the transfer from the purge and trap device. The carbon traps should be changed a minimum of every three months.

Restek RTX-624 Column: 20m x 0.18mm ID, 1.0um film thickness, or equivalent.

EST Encon purge and trap concentrator with 5mL sparge vessel, or equivalent.

EST Centurion Autosampler, or equivalent.

Supelco Vocarb 3000 trap or equivalent. Other traps may be used as long as the target compounds can be detected at the required quantitation limit and the IDOC requirements are met.

6.2 Analytical Data System / Software / Hardware

Chemstation software is used on a Windows-based PC to schedule and acquire data. CHROM software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's LIMS system (i.e., TALS). CHROM software has the capability of processing stored GC data by recognizing a GC peak within any given retention time window and comparing the retention time of the sample to the retention times of the standards analyzed under the same conditions. The software also allows calculation integration of the peak responses, response factors, construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

6.3 Volumetric Labware

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*. Refer to Attachment 8 for labware cleaning procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Verification Criteria
Volumetric Flasks (Class A)	25mL 50mL	QUANTITATIVE	Preparing Standards; Preparing Dilutions	None	None (Purchased Class A)
VOA Vials	40mL	QUANTITATIVE	Sample Collection and Analysis	Per Lot	Accuracy = 2% Precision = 1%
Gas-Tight Syringes	Various	QUANTITATIVE	Standard Preparation and Spiking	None	None (If received with COA)
Mini-Nert Vials	Various	Qualitative	Standard Storage	None	None

6.4 Lab Supplies

pH paper

Residual Chlorine Check Strips – starch iodide strips; provide a quick and easy way to verify if the sample was dechlorinated properly. Store in original, capped container and use within the manufacturer's expiration date.

Detergent – Liquinox used for washing non-disposable labware.

6.5 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are packaged in a kit which contains:

- 3x 40mL VOA vials containing ascorbic acid granules (used for dechlorination)
- 1 HCl dropper containing 1mL 1:1 HCl (used for preservation)

The kits are purchased with a Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

Unless listed elsewhere in this SOP, the expiration dates given below apply.

7.1.1 The expiration date for unopened standards and reagents is the manufacturer's expiration date.

7.1.2 The expiration date for opened stock reagents is the manufacturer's expiration date or 5 years from the date opened, whichever is sooner.

7.1.3 The expiration date for opened stock standards is the manufacturer's expiration date or 1 month from the date opened, whichever is sooner.

7.1.4 The expiration date for prepared reagents is 6 months from the date prepared or the expiration date of the parent reagent, whichever is sooner.

7.1.5 The expiration date for prepared standards is 1 month from the date prepared or the expiration date of the parent standard, whichever is sooner.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Methanol must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Laboratory Reagent Water – ASTM Type II, obtained from on-site well

Methanol – Purge and Trap grade
Storage: Flammable Cabinet

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached to the standard in TALS.

Refer to Attachment 6 for standard preparation information.

Unopened source standards must be stored at manufacturer's recommended conditions. After opening, standards are stored in the freezer at < -10°C.

8.0 Sample Collection, Preservation, Shipment, and Storage

Aqueous samples are routinely collected in triplicate. Two vials are retained for analysis and the third vial is used to check the sample pH and for the presence of residual chlorine. This "sacrifice" vial should not be used for analysis unless all other vials have been consumed. If the "screening vial" is used for analysis, a Nonconformance Memo (NCM) must be initiated.

Samples are routinely collected with no headspace in 40mL vials equipped with Teflon-lined caps. The samples are dechlorinated with 25mg of ascorbic acid and acidified with about 1.0mL of 1:1 HCl per 40mL of sample at the time of collection. The preservative should be sufficient to achieve a sample pH of less than 2. The dechlorination agent should be sufficient to remove residual chlorine from the sample.

Samples must be iced at the time of collection and refrigerated at 0-6°C (less than 6°C with no frozen samples) in the lab until analysis. Samples must be analyzed within 14 days of collection. If the samples are unpreserved or if the pH >2, the samples must be analyzed within 24 hours of collection.

Note: If Total Trihalomethanes (THM) are the only analytes requested, the acid may be omitted and the samples may be dechlorinated with 4mg of sodium thiosulfate per 40mL of sample at the time of collection.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives and/or de-chlorination agents. NCMs must be initiated for samples that are received containing headspace.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatile Samples* for additional information.

8.1 Preservation Checks

These checks can be performed upon receipt or prior to preparation.

8.1.1 Mix the sample by inverting. Using a 100uL syringe, withdraw approximately 50uL of sample through the septa.

8.1.2 Dispense a small amount of sample onto a piece of narrow range pH paper and note the pH. Record the pH in the PRESERV_CHK method in TALS.

If the pH is greater than or equal to 2, initiate an NCM noting that the pH was outside of the preservation requirements.

Note: If the pH is greater than or equal to 2, a 24-hour holding time is enacted. Notify the Project Manager via a NCM if the 24-hour holding time is not met.

8.1.3 Dispense remaining sample onto a piece of starch iodide paper and note the color change of the paper.

If the paper turns blue or black, residual chlorine is present. Record whether the sample contains residual chlorine in the PRESERV_CHK method in TALS. Initiate a Nonconformance Memo if the sample contains residual chlorine.

8.1.4 Repeat steps 8.1.1 through 8.1.3 individually for each sample. Check the pH, then record the pH result in the TALS batch; check for residual chlorine, then record the residual chlorine result in the TALS batch – prior to proceeding to the next sample.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An analytical batch consists of up to 20 environmental samples and the associated QC items analyzed together within a 12 hour period. The minimum QC items required for each batch are: a method blank, a laboratory control sample (LCS), a low-level LCS (spiked at the reporting limit), and a matrix spike (MS), and a matrix spike duplicate (MSD)

If there is insufficient sample to perform the required MS and/or MSD, the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample is defined as receiving less than 4 vials.

Note: The LCS must be analyzed in duplicate at least once a quarter.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Note: The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. Therefore, if analyzing drinking water samples by EPA 524.2, an LCS at the RL must also be included in the required batch QC.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Instrument QC

The term “clock time” or “analytical clock” refers to the amount of time that can pass before additional instrument QC items must be performed. The analytical clock begins with the injection of the BFB, and all subsequent injections must be completed before the

clock time expires – at which point new instrument QC is performed and a new clock is initiated.

The clock time for EPA 524.2 is defined as 12 hours.

Note: Due to instrument configurations employing dual concentrators, most of the laboratory instruments can analyze more than 20 injections within the designated clock times. An analytical batch is still defined as 20 field samples; therefore, if more than 20 field samples are analyzed within a clock, additional batch QC is required (i.e., another method blank, LCS, and MS/MSD must be performed).

9.2.1 Tune Check

Inject 1uL of the 25ng/uL BFB standard.

Note: The analysis may be performed using purge and trap or by direct injection of the BFB standard. Mass spectrometer conditions must be the same as for the standard and sample analyses. The temperature programs may be different to allow for timely elution of BFB.

Evaluate the spectrum of the BFB peak. Test the apex of the peak first against the acceptance criteria. If the apex does not meet the criteria, evaluate the scans plus one and minus one scan from the apex. An average spectrum across the peak may also be evaluated against the criteria. If background subtraction is required, choose a spectrum at least ten scans before the elution of the peak for background.

TUNING AND MASS CALIBRATION ACCEPTANCE CRITERIA	
m/e	**Abundance Criteria**
50	15-40% of mass 95
75	30-80% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	< 2% of mass 174
174	Greater than 50% of mass 95
175	5-9% of mass 174
176	> 95% but < 101% of mass 174
177	5-9% of mass 176

Note: The p-BFB analysis must meet the criteria before any standards or samples may be analyzed. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. If there is any question about whether the BFB passes the criteria, contact the supervisor immediately before proceeding.

If the p-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the p-

BFB analysis, other corrective measures may include remaking the p-BFB standard, cleaning the instrument ion source, etc. Additionally, the chromatogram of the tuning analysis should be checked for acceptable baseline and the p-BFB peak should be symmetrical.

9.2.2 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 3 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

Standard Level	Concentration (ug/L)
1	0.5
2	1.0
3	2.0
4	5.0
5	10
6	20
7	50
8*	100

*Used for TTHMs only.

Refer to Attachment 6 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

Note: EPA 524.2 requires a minimum of a 3-point calibration curve for a 20 fold concentration range, a 4-point calibration curve for a 50 fold concentration range, and a 5-point calibration curve for a 100 fold concentration range.

9.2.2.1 ICAL Criteria

The relative standard deviation of the calibration standards must be <20% for the initial calibration curve to be acceptable.

If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted.

9.2.3 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The ICV must be within 30% to be acceptable.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to level 6 of the ICAL. Refer to Attachment 6 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

Note: The LCS may be used to satisfy the ICV requirement if it is prepared from a second source and meets the criteria outlined above.

9.2.4 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately following the initial calibration. Continuing calibration blanks are analyzed immediately following the continuing calibration verification (CCV).

Initial and continuing calibration blanks must be <1/2RL to be acceptable.

9.2.5 Continuing Calibration Verification

The initial calibration curve must be verified at the beginning of each clock with a mid-level standard.

The CCV must be within 30% to be acceptable.

The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to level 6 of the ICAL. Refer to Attachment 6 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.6 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. Fluorobenzene is the internal standard.

Prior to analysis, this internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. A concentration of 10ug/L is used.

The response of the ISTD in the ICV/CCV must be within 30% of the response of the ISTD in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

The response of the ISTD in the samples and batch QC items must be within 30% of the response of the previous CCV. If the response is outside of this range, corrective action must be taken.

9.2.7 Surrogate

This procedure uses surrogates to evaluate the analytical process. 1,2-Dichlorobenzene-d4 and 4-Bromofluorobenzene are the surrogates.

Prior to analysis, this surrogate is added to all samples and QC items. The concentration of the surrogate is the same in all field samples and QC samples. A concentration of 10ug/L is used.

The percent recovery of the surrogate in all field samples and QC samples must be within the limits listed in the Method Limit Groups (MLGs) in TALS. If the percent recovery is outside of this range, the analysis of the sample must be repeated. Repeated failure of the surrogate percent recovery may indicate instrumentation problems.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

Composite samples can be prepared using the guidance provided in SOP SA-QA-015: *Compositing, Homogenization, and Segregation of Samples*.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for additional information.

10.2 QC Sample Preparation

10.2.1 Method Blank – The method blank is prepared as follows: Fill a 50mL volumetric with reagent water. Add 50uL of ISSU. Invert flask three times and transfer contents to a 40mL VOA vial (containing HCl preservative) with no headspace. Place on instrument to be analyzed.

10.2.2 Laboratory Control Sample – The LCS is prepared as follows: Fill a 50mL volumetric with reagent water. Add 20uL of Mega Mix, 20uL of Additional Mix, 50uL of ISSU, and 160uL

of MeOH. Invert flask three times and transfer contents to a 40mL VOA vial (containing HCl preservative) with no headspace. Place on instrument to be analyzed.

10.2.3 Low-Level Laboratory Control Sample – The LLCS is prepared as follows: Fill a 50mL volumetric with reagent water. Add 0.5uL of Mega Mix, 0.5uL of Additional Mix, 50uL of ISSU, and 199uL of MeOH. Invert flask three times and transfer contents to a 40mL VOA vial (containing HCl preservative) with no headspace. Place on instrument to be analyzed.

10.2.4 Matrix Spike – Matrix spikes are prepared as follows: Spike 17.2uL of Mega Mix, 17.2uL of Additionals Mix, 43uL of ISSU, and 137.6uL of MeOH into a 40mL VOA vial containing the sample designated for the MS and MSD. Place in the instrument to be analyzed.

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Note: The drinking water methods are prescriptive. For this reason, items such as purge volume, purge gas, purge time, carrier gas, etc. must match the EPA method.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

Note that the MS must be set to monitor ions between 35 and 260amu with a scan rate of 1 second or less. The purge time must be 11 minutes. All other parameters may be changed to optimize the system.

Column: Restek RTX-624 0.18mm x 20m x 1.0um, or equivalent

Helium carrier gas flow rate: 0.5mL/min (constant flow)
Inlet Pressure: 15.8 psi
Total Flow: 28.2mL/minute
Split Ratio: 50:1 (Routine and Tune Check)
Split Ratio: 25:1 (UCMR List 1 Compounds)
Split Flow: 25mL/min
Gas Saver: 20.0 mL/min @ 2.00 min

Routine Targets and UCMR List 1	**BFB Tune Check**
Initial column temperature: 40°C for 1min	Initial column temperature: 50°C for 1min
Column temperature program 1: 17°C/min	Column temperature program 1: 17°C/min
Final column temperature: 200°C for 7min	Final column temperature: 200°C for 7min
Run Time: 11.41 minutes	Run time: 8.82 minutes

Injector temperature: 250°C
Mass range: 35-260amu
Solvent Delay: 0.90 Minutes
Threshold: 150 Sample #: 2 A/D Samples: 4
MS Quad Temperature: 150°C MS Source Temperature: 250°C
EM Absolute: TRUE
Resulting EM Voltage: EM voltage set at AUTOTUNE + 200
Tune File: TUS.U for instrument MSS
Tune File: TUU.U for instrument MSU

Purge and Trap Instrument Conditions

Purge Time: 11 min
Purge Temperature: Ambient
Desorb Time: 0.5 min
Desorb Temperature: 250°C
Bake Time: 8 min at 260°C
Purge Flow: Approximately 35 mL/min. Adjust to maximize response of chloromethane and bromoform.
Valve Temperatures: 150°C
Transfer Line: 150°C

10.3.2 Internal Standard (ISTD)

Prior to analysis, 43uL of ISSU must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. A concentration of 10ug/L is used.

10.3.3 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.2. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.3, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5.

10.3.4 Sample Analysis

Remove the samples from the refrigerator and allow them to come to room temperature.

The sample must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The default procedure is to exclude QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in the clock.

10.3.5 Example Analytical Sequence

See Attachment 1 for an example analytical sequence.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data.* This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data review and reporting must be performed in accordance with SA-QA-002: *Data Generation and Review.*

11.1 Qualitative Analysis of Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1 Two criteria must be met in order to identify a target-compound.

1) elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound.

$$RRT = \frac{retention\ time\ of\ the\ target\ compound}{retention\ time\ of\ the\ associated\ internal\ standard}$$

2) correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. These ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within +/- 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.1.5 MS/MSD Evaluation

If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data is flagged.

11.1.2 Evaluation of Tentatively Identified Compounds (TICs)

Refer to Attachment 11 of SOP SA-QA-008: *Evaluation of Chromatographic Data* for the laboratory's TIC processing procedures.

11.1.3 Dilutions

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

11.1.4 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.1.5 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met, the Department Supervisor must be contacted immediately.

Benzene, toluene, ethylbenzene, and the xylenes are generally present together in samples and indicate the presence of gasoline

m/p-Xylenes are generally higher than o-xylene

Hydrocarbons present is samples containing gasoline generally contain mass 43 and may co-elute with target analytes with mass 43 as the quant or confirmation ion or may skew the spectrum of a compound with mass 43 as part of the spectrum.

Cis- isomers are generally more prevalent than the trans- isomers

Pay particular attention to the retention time of isomer because the only way to positively identify them is by retention time. The isomers are:

1,1-dichloroethane and 1,2-dichloroethane
1,1-dichloroethene, cis-1,2-dichloroethene, and trans-1,2-dichloroethene
1,1,1-trichloroethane and 1,1,2-trichloroethane
ethyl benzene, m/p-xylene, and o-xylene
1,3-dichlorobenzene, 1,4-dichlorobenzene, and 1,2-dichlorobenzene
1,1-dichloropropene, cis-1,2-dichloropropene, and trans-1,2-dichloropropene
2-chlorotoluene and 4-chlorotoluene
1,2,3-trichlorobenzene and 1,2,4-trichlorobenzene
1.3.5-trimethylbenzene and 1,2,4-trimethylbenzene
4-methyl-2-pentanone (MIBK) and 2-hexanone
n-butylbenzene, sec-butylbenzene, tert-butylbenzene, and isopropylbenzene

Higher chlorinated alkanes and alkenes may have lower chlorinated alkanes or alkenes present due to degradation. The following table lists some common chlorinated compounds and their degradation products. Look for the degradation product(s) when the concentration of the compound in the left column is present at high concentrations.

Analyte	Degration Product
1,1,2,2-tetrachloroethane	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) vinyl chloride 1,1,2-trichloroethane (1,1,2-TCA) 1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,2-trichloroethane (1,1,2-TCA)	1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,1-trichloroethane (1,1,1-TCA)	1,1-dichloroethene (1,1-DCE) 1,1-dichloroethane (1,1-DCA) Chloroethane
Carbon tetrachloride	Chloroform Methylene chloride Chloromethane
Tetrachloroethene (PCE) (PCE = perchloroethylene which is a common name for tetrachloroethene)	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) Chloroethene
1,2,4-trichlorobenzene	1,4-dichlorobenzene (1,4-DCB) 1,2-dichlorobenzene (1,2-DCB) Chlorobenzene

Trihalomethanes are formed when water from a natural source (river, well, etc.) is chlorinated. Usually, THM will be present in the relative concentrations as follows:
chloroform >> dichlorobromomethane > dibromochloromethane >> bromoform.

11.1.6 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCLs associated with this procedure are given in Attachment 8. Notify the PM immediately via a Nonconformance Memo if any sample contains a detection above these levels.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor

Note: This calculation assumes all applicable unit correction factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

In addition to the requirements in SOP SA-QA-007, EPA 524.2 also requires that MDL studies be performed over multiple days.

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Note: The IDOC must meet 80-120% recovery and less than 20% RSD.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess reagents – Dispose as outlined below.

Material	Treatment	Disposal Destination
Methanol	None	Flammable Waste Drum
Standards	None	Flammable Waste Drum
Hydrochloric Acid and Solutions	Neutralize	Sink

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- SOP SA-VO-001*: Preparation, Storage, and Screening of Volatiles Samples*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- US EPA 524.2 Revision 4.1: *Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry*; 1995

16.0 Method Modifications

16.1 The reference method was written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure.

16.2 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each batch of samples. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory defaults to 50-150%. These criteria are required for THMs, as specified in the Disinfection By-Product Rule.

16.3 The laboratory has incorporated the minimum batch QC items as outlined in Section 9.1. Some additional QC items are routinely performed above those required in the EPA 524.2 reference method (i.e., MS/MSD and/or LCS/LCSD) to satisfy common regulatory and/or client requests for precision data and/or to facilitate scheduling and data evaluation.

16.4 Due to the volatile nature of the analytes tested, the laboratory sacrifices a vial to be used for pH check, residual chlorine verification, and screening. The laboratory applies the pH and residual chlorine values identified on this vial to the remaining vials submitted for that sample (e.g., if the pH of the tested vial is acceptable, the remaining vials for that sample are assumed to be acceptable). The practice of checking pH prior to analysis allows for re-adjustment of holding times based on the preservation of the sample, as outlined in Attachment 2.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Standard Preparation
Attachment 6: List of Regulated Analytes and MCLs
Attachment 7: Quant Ions
Attachment 8: Glassware Cleaning Procedures

Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary

Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Analytical Sequence

Description	Comments
Blank	
Tune	12-hour clock begins with injection of the tune
Initial Calibration	
ICV	Second Source
ICB	
Samples & Batch QC Items	Not to exceed 12 hours; last injection must occur before 12 hours from BFB injection
Tune	12-hour clock begins with injection of the tune
CCV	20ug/L
CCB	
Samples & Batch QC Items	Not to exceed 12 hours; last injection must occur before 12 hours from BFB injection
Tune	12-hour clock begins with injection of the tune
CCV	20ug/L
CCB	

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation[1]	Thermal Preservation	Holding Time[3]
Water	3 x 40mL VOA vial	40mL	40mL	Ascorbic Acid	1:1 HCl	0-6°C[2]	pH<2: 14 days pH>2: 24 hours
Water (TTHM Only)	3 x 40mL VOA vial	40mL	40mL	Sodium Thiosulfate	Not Applicable	0-6°C[2]	14 days

[1]Samples must be dechlorinated prior to acidification.
[2]Samples are collected on ice and maintained at <6°C with no frozen samples.
[3]Holding time is from sample collection to analysis.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Clock Time	12 hours	Clock time starts with the injection of the BFB. Analysis of samples and QC items must conclude within expiration of clock time. Subsequent analysis requires new BFB.	Not applicable
Tune Standard (BFB)	At beginning of each clock	Refer to Section 9.2.1.	- Perform instrument maintenance - Re-tune.
Initial Calibration (ICAL) - Minimum 3 points	Upon instrument set-up, and after unsuccessful CCV	%RSD < 20% If %RSD > 20%, use curve fit w/ r^2>0.990.	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Initial Calibration Verification (ICV) - Second Source	After each ICAL	%RSD < 30%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Continuing Calibration Verification (CCV)	After BFB	%RSD < 30%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate

QC Item	Frequency	Criteria	Corrective Action
Calibration Blank (ICB/CCB)	After ICV and every CCV	<1/2RL	Refer to SOP SA-QA-017
Internal Standards (ISTD)	Spiked in all CCVIS, samples, and batch QC items	CCVIS: - Area within 30% of CCV in ICAL. Samples & batch QC items: - Area within 30% of previous CCVIS.	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	Spiked in all samples and batch QC items.	70-130%	-Evaluate chromatogram, spectra, and integrations -Reanalyze sample, if sufficient sample available
Analytical Batch Definition	Analyzed together w/in 12-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per analytical batch	<1/2RL	Refer to SOP SA-QA-017
Laboratory Control Sample (LCS)	One per analytical batch	70-130% Rec	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per analytical batch, when insufficient sample is provided for MS/MSD	70-130% Rec; <30%RPD	Refer to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
Low-Level Laboratory Control Sample (LLCS)	One per analytical batch	50-150% Rec	Refer to SOP SA-QA-017
Matrix Spike (MS)	One per analytical batch (If additional sample volume provided by client.)	70-130% Rec	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	One per analytical batch (If additional sample volume provided by client.)	70-130% Rec; <30%RPD	Refer to SOP SA-QA-017
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	80-120% Rec; <20% RPD	Refer to SOP SA-QA-006 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL) - Must be performed over multiple days	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

QC Item	Frequency	Criteria	Corrective Action
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Injector Port							X	Replace septum, sleeve, inlet seal, and washer (Recommend every 2 weeks)
Sparge Tubes							X	Clean (Recommend every 3 months)
Column							X	Change column (Recommend annually)

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation,

the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Attachment 5:
Standard Preparation

Purchased Standards

Mega Mix, 2000 ug/mL – NSI Solutions

Mega Mix 2 (Secondary Standard), 2000ug/mL – Restek

Gases Mix, 2000ug/mL – Supelco

Gases Mix 2 (Secondary Standard), 2000ug/mL – NSI

California Oxygenates Mix 1, 2000-10000 ug/mL – Restek

California Oxygenates Mix 2 (Secondary Standard), 2000-10000 ug/mL – O2Si

Volatile Organics Calibration Mix, 5000ug/mL – Restek

Ketone 2 (Secondary Standard) 2000ug/mL – Supelco

Freon, 2000ug/mL – O2SI

Freon 2 (Secondary Standard), 2000ug/mL – Ultra Scientific

Internal Standard and Surrogate Mix, 2000 ug/mL

BFB (Tune) - NSI

Prepared Standards

524 Mega Mix (Working Standard), 50-150ug/mL – Prepared by adding 250uL of Mega Mix and 250uL of Gases Mix to 10mL of methanol.

524 Mega Mix 2 (Secondary Working Standard), 50-150ug/mL – Prepared by adding 250uL of Mega Mix 2 and 250uL of Gases Mix 2 to 10mL of Methanol.

524 Additions (Working Standard), 40-200ug/mL – Prepared by adding 200uL of California Oxygenates Mix 1, 200uL of Freon, and 200uL of Volatile Organics Calibration Mix to 10mL of methanol.

524 Additions 2 (Secondary Working Standard), 40-200ug/mL – Prepared by adding 200uL of California Oxygenates Mix 2, 200uL of Freon 2, and 500uL of Ketones 2 to 10mL of methanol.

524 ISSU (Working Standard), 10ug/mL – Prepared by adding 125uL of Internal Standard and Surrogate Mix to 25mL of methanol

BFB (tune), 25ng/uL – Prepared by adding 125uL of BFB to 10mL of methanol.

ICAL Standards

Stock/Mix	1	2	3	4	5	6	7	8
	Aliquot to prepare CAL standard (uL)							
524 Mega Mix	0.5	1.0	2.0	5.0	10	20	50	100
524 Additionals	0.5	1.0	2.0	5.0	10	20	50	100
524 ISSU	50	50	50	50	50	50	50	50
Methanol	199	198	196	190	180	160	100	---
Volume of water (mL)	50	50	50	50	50	50	50	50
Concentration								
Target Compounds (ng)	2.5	5	10	25	50	100	250	500
Internal Standards (ng)	50	50	50	50	50	50	50	50

Note: All initial and continuing calibration standards are prepared in 50mL volumetric flasks and then poured into 40mL VOA vials, containing HCl preservative, for analysis on the instrument.

Attachment 6:
List of Regulated Analytes and MCLs

Analyte	MCL (ug/L)
Benzene	5
Carbon tetrachloride	5
Chlorobenzene	100
1,2-Dichlorobenzene	600
1,4-Dichlorobenzene	75
1,2-Dichloroethane	5
1,1-Dichloroethene	7
Cis-1,2-Dichloroethene	70
Trans-1,2-Dichloroethene	100
1,2-Dichloropropane	5
Ethylbenzene	700
Methylene chloride	5
Styrene	100
Tetrachloroethene	5
Toluene	1000
1,2,4-Trichlorobenzene	70
1,1,1-Trichloroethane	200
1,1,2-Trichloroethane	5
Trichloroethene	5
Vinyl chloride	2
Total Xylenes (Sum of o-xylenes and m/p-Xylenes)	10000
Trihalomethanes, total (Sum of chloroform, bromoform, dibromochloromethane, and dibromochloromethane)	100

Attachment 7:
Quant Ions

Compound	CAS	ISTD	Quant Ion	Secondary Ions	
1,1,1,2-Tetrachloroethane	630-20-6	1	131	133	119
1,1,1-Trichloroethane	71-55-6	1	97	99	61
1,1,2,2-Tetrachloroethane	79-34-5	1	83	85	168
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	1	101	51	43
1,1,2-Trichloroethane	79-00-5	1	97	83	99
1,1-Dichloroethane	75-34-3	1	63	65	83
1,1-Dichloroethene	75-35-4	1	61	96	98
1,1-Dichloropropene	563-58-6	1	75	110	77
1,2,3-Trichlorobenzene	87-61-6	1	180	182	109
1,2,3-Trichloropropane	96-18-4	1	110	112	
1,2,4-Trichlorobenzene	120-82-1	1	180	182	145
1,2,4-Trimethylbenzene	95-63-6	1	105	120	77
1,2-Dibromo-3-Chloropropane	96-12-8	1	75	157	155
1,2-Dichlorobenzene	95-50-1	1	146	148	111
1,2-Dichlorobenzene-d4 (Surrogate)	2199-69-1	1	152	115	150
1,2-Dichloroethane	107-06-2	1	62	49	64
1,2-Dichloropropane	78-87-5	1	63	76	65
1,3,5-Trimethylbenzene	108-67-8	1	105	120	77
1,3-Dichlorobenzene	541-73-1	1	146	148	111
1,3-Dichloropropane	142-28-9	1	76	78	41
1,4-Dichlorobenzene	106-46-7	1	146	148	111
2,2-Dichloropropane	594-20-7	1	77	41	
2-Butanone (MEK)	78-93-3	1	43	72	
2-Chlorotoluene	95-49-8	1	91	126	63
2-Hexanone	591-78-6	1	43	85	100
2-Methyl-2-propanol (TBA)	75-65-0	1	59	41	43
4-Bromofluorobenzene (Surrogate)	460-00-4	1	95	174	176
4-Chlorotoluene	106-43-4	1	91	126	63
4-Isopropyltoluene	99-878-6	1	119	134	91
4-Methyl-2-pentanone (MIBK)	108-10-1	1	43	85	100
Acetone	67-64-1	1	43	58	
Benzene	71-43-2	1	78	50	51
Bromobenzene	108-86-1	1	77	156	158
Bromoform	75-25-2	1	173	171	174
Bromomethane	74-83-9	1	94	96	79
Carbon tetrachloride	56-23-5	1	117	119	121
Chlorobenzene	108-90-7	1	112	77	51
Chlorobromomethane	74-97-5	1	49	130	

Compound	CAS	ISTD	Quant Ion	Secondary Ions	
Chlorodibromomethane	124-48-1	1	129	127	131
Chloroethane	75-00-3	1	64	66	
Chloroform	67-66-3	1	83	85	47
Chloromethane	74-87-3	1	50	52	
cis-1,2-Dichloroethene	156-59-2	1	61	96	98
cis-1,3-Dichloropropene	10061-01-5	1	75	77	110
Dibromomethane	74-95-3	1	93	174	95
Dichlorobromomethane	75-27-4	1	83	85	129
Dichlorodifluoromethane	75-71-8	1	85	87	101
Ethylbenzene	100-41-4	1	91	106	51
Ethylene Dibromide	106-93-4	1	107	109	
Fluorobenzene (Internal Standard)	17060-07-0	1	96	70	50
Hexachlorobutadiene	87-68-3	1	225	223	190
Isopropyl ether	108-20-3	1	45	59	87
Isopropylbenzene	98-82-8	1	105	120	77
Methyl tert-butyl ether	1634-04-4	1	73	57	
Methylene Chloride	75-09-2	1	49	84	86
m-Xylene & p-Xylene	136777-61-2	1	91	106	77
Naphthalene	91-20-3	1	128	102	51
n-Butylbenzene	104-51-8	1	91	92	134
Nitrobenzene	98-95-3	1	96	70	50
N-Propylbenzene	103-65-1	1	91	120	65
o-Xylene	95-47-6	1	91	106	77
sec-Butylbenzene	135-98-8	1	105	134	91
Styrene	100-42-5	1	104	78	103
Tert-amyl methyl ether	994-05-8	1	73	43	87
Tert-butyl ethyl ether	637-92-3	1	59	87	57
tert-Butylbenzene	98-06-6	1	119	91	134
Tetrachloroethene	127-18-4	1	166	164	168
Toluene	108-88-3	1	91	92	65
trans-1,2-Dichloroethene	156-60-5	1	61	96	98
trans-1,3-Dichloropropene	10061-02-6	1	75	77	110
Trichloroethene	79-01-6	1	130	95	132
Trichlorofluoromethane	75-69-4	1	101	103	105
Vinyl chloride	75-01-4	1	62	64	

Attachment 8: Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES

VOLATILES DEPARTMENT

1. Rinse glassware 3 times thoroughly with DI water.
2. Place glassware, top-down, within storage rack and allow to air dry.
3. If glassware was used to prepare waste sample, use FL-70 and water to scrub glassware and follow previous steps.

FVM008:05.14.14:2

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

18.0 Revision History

Summary of Changes from Previous Revision:

- Minor grammatical and/or editorial edits.
- Clarified section on sample collection containers to include reference to kit containing ascorbic acid and HCl. Section 6.5
- Added requirement to pour prepared method blank, LCS/LCSD, and LLCS into VOA vials containing HCl. This change stems from a 2015 MA DEP Data Review. Section 10.2.3
- Added requirement to pour prepared initial and continuing calibration standards into VOA vials containing HCL. This change stems from a 2015 MA DEP Data Review. Attachment 5

ALS Environmental

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QUALITY ASSURANCE MANUAL

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QUALITY ASSURANCE MANUAL

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TABLE OF CONTENTS

QA MANUAL CROSS REFERENCE TABLE

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14	5.9	2/5.9
21	5.10	2/5.10

1) Introduction and Scope

The purpose of this Quality Assurance Manual is to outline the quality system for the Simi Valley location of ALS Environmental (ALS Group USA Corp. dba ALS Environmental). ALS Environmental is a professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, air, and other material. Refer to Appendix J for a list of analytical capabilities specific to the Simi Valley location and corresponding accreditation status.

Quality Control (QC) procedures are used to continually assess performance of the laboratory and quality systems. ALS Environmental maintains control of analytical results by adhering to written standard operating procedures (SOPs), using analytical control parameters with all analyses, and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data. Appendix H includes a list of data qualifiers and acronyms.

This QAM is applicable to the facility listed on the title page and the off-site extraction facility located at 2360 Shasta Way, Unit G, Simi Valley California.

The information in this QAM has been organized according to requirements found in the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards (2003 and 2009), the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, USEPA, 2001; and *General Requirements for the Competence of Testing and Calibration Laboratories*, ISO/IEC 17025:2005.

2) Organization

2.1 Laboratory Organizational Structure

ALS Environmental – Simi Valley is legally identifiable as ALS Group USA, Corp., dba ALS Environmental. ALS Group USA Corp. is a component of ALS Limited, a publicly held Australian company. The ALS global website may be referred to for corporate ownership information (www.alsglobal.com/Our-Company). Organizational charts detailing the operational structure and reporting relationships in the laboratory are provided in Appendix B.

2.2 Avoiding Conflict of Interest through Organizational Structure

2.2.1 Through application of the policies and procedure outlined in this QA Manual and use of a defined organizational structure, the laboratory assures that it is impartial and that personnel are free from undue commercial, financial, or other undue pressures that might influence their technical judgment.

2.2.2 Policies are in place to prevent outside pressures or involvement in activities that may affect competence, impartiality, judgment, operational integrity, or the quality of the work performed at the laboratory.

2.2.3 Management and technical personnel have the authority and resources to carry out their duties and have procedures to identify and correct departures from the laboratory's management system.

2.2.4 Personnel understand the relevance and importance of their duties as related to the maintenance of the laboratory's management system. Ethics and data integrity procedure ensure that personnel do not engage in activities that diminish confidence in the laboratory's capabilities. Procedures and policies are also established to ensure confidentiality is maintained.

3) Management

The purpose of the QA program at ALS Environmental is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality.

3.1 Quality Policy Statement

The policy at ALS is to use good professional practices, to maintain quality, to uphold the highest standard of service, and to operate in accordance with these requirements and those of regulatory agencies, accrediting authorities, and certifying organizations. We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory. Laboratory management is committed to ensuring the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set forth in the *SOP for Laboratory Ethics and Data Integrity* (CE-GEN001) and in this Quality Assurance Manual (QAM). ALS Environmental is committed to operate in accordance with these requirements and those of regulatory agencies, accrediting authorities, and certifying organizations. The laboratory also strives for improvement through varying continuous improvement initiatives and projects.

Quality Management Systems are established, implemented and maintained by management. Policies and procedures are established in order to meet requirements of accreditation bodies and applicable programs as well as client's quality objectives. The laboratory's management is committed to complying with the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards (2003 and 2009 NELAP/TNI standards), ISO/IEC 17025:2005, and the Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory is involved. All personnel involved with environmental testing and calibration activities must familiarize themselves with the quality documentation and implement the policies and procedures in their work.

3.2 Quality Management Systems

The laboratory has developed a Quality Management System to ensure all products and services meet our client's needs. The system is implemented and maintained by the Quality Assurance Manager (QA Manager) with corporate oversight by the Corporate Quality Assurance Manager (CQAM). These systems are based upon ISO 17025:2005 standards, upon which fundamental programs (AIHA, TNI/NELAP, and DoD QSM) are based. Implementation and documentation against these standards are communicated in corporate policy statements, this QAM, and SOPs. Actual procedures, actions and documentation are defined in both administrative and technical SOPs. Figure 3-1 shows the relationships of the quality systems and associated documentation. Quality systems include:

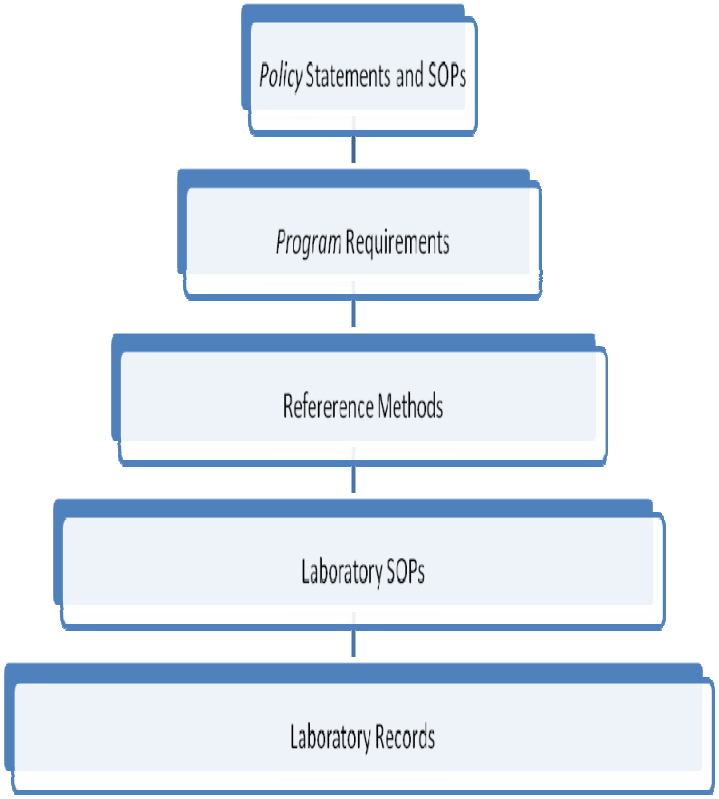
- Standard Operating Procedures
- Sample Management and Chain of Custody procedures
- Statistical Control Charting
- Standards Traceability
- Ethics Training
- Document Control
- Corrective Action Program
- Management Reviews
- Demonstration of Capability

The effectiveness of the quality system is assessed in several ways, including:

- Internal and External Audits covering all aspects of the organization
- Annual Management Reviews
- Analysis of Customer Feedback
- Internal and External Proficiency Testing

Figure 3-1

Relationships of Quality Management Systems and Documentation



3.3 Technical Elements of the Quality Assurance Program

The laboratory's technical procedures are based upon procedures published by various agencies or organizations (See Section 23). The Quality Assurance Program provides laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained by the QA department. This includes method approvals and audit

administration. In addition, internal audits are performed to assess compliance with policies and procedures. SOPs are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix I and SOPs in Appendix G.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, technical training and preventive maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

3.4 Professional Conduct

One of the most important aspects of the success of ALS Environmental is the emphasis placed on the integrity of the data provided and the services rendered. This success is reliant on both the professional conduct of all employees within ALS Environmental as well as established laboratory practices.

To promote quality, ALS Environmental requires certain standards of conduct and ethical performance among employees. The following examples of documented ALS Environmental policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action.
- Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible.
- It is the responsibility of all ALS Environmental employees to safeguard sensitive company information, client data, records, and information; and matters of national security concern should they arise. The nature of our business and the well-being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action. All employees sign a confidentiality agreement upon hire to protect the company and client's confidentiality and proprietary rights.

3.5 Prevention and Detection of Improper, Unethical or Illegal Actions

It is the intention of ALS Environmental to proactively prevent and/or detect any improper, unethical or illegal action conducted within the laboratory.

This is performed by the implementation of a program designed for not only the detection but also prevention. Prevention consists of educating all laboratory personnel of their roles and duties as employees, company policies, inappropriate practices, and their corresponding implications as described here.

In addition to education, appropriate and inappropriate practices are included in SOPs such as manual integration, data review and specific method procedures. Electronic and hardcopy data audits are performed regularly, including periodic audits of chromatographic electronic data. Requirements are described in the *SOP for Internal Audits* (CE-QA001) and details are listed in laboratory administrative SOPs. All aspects of this program are documented and retained on file according to the company policy on record retention.

The *SOP for Laboratory Ethics and Data Integrity* (CE-GEN001) also contains information on the ALS Environmental ethics and data integrity program, including mechanisms for reporting and seeking advice on ethical decisions.

3.6 Laboratory Data Integrity and Ethics Training

New employees are given a QA and Ethics orientation within the first month of hire. On an ongoing basis, all employees receive annual ethics refresher training. Topics covered are documented in writing and all training is documented. It is the responsibility of the QA Manager to ensure that the training is conducted as described.

Key topics covered are the organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues and record keeping. Training includes discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation.

Data integrity training provides assurance that a highly ethical approach to testing is a key component of all laboratory planning, method implementation, and training. There are four elements to the laboratory's procedures for data integrity. These include:

1) Data integrity training (conducted initially and at least annually);
2) Signed data integrity documentation for all employees;
3) In-depth periodic monitoring of data integrity;
4) Data integrity procedure documentation (*SOP for Laboratory Ethics and Data Integrity* (CE-GEN001)).

There is specific emphasis on the importance of proper written narration on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially deficient. A signature attestation sheet of data integrity training including their understanding of their obligations related to data integrity and as specified in the training is generated for attendees and maintained on file for review. Trainees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, or civil/criminal prosecution.

The training session includes many concepts and topics, numerous examples of improper actions (defined by DoD as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional), legal and liability implications (company and personal), causes, prevention, awareness, and reporting mechanisms.

3.7 Management and Employee Commitment

ALS Environmental makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the *SOP for Laboratory Ethics and Data Integrity* (CE-GEN001). This includes:

- ALS Environmental Open Door Policy – Employees are encouraged to bring any work related problems or concerns to the attention of local management or their Human Resources representative. However, depending on the extent or sensitivity of the concern, employees are encouraged to directly contact any member of upper management.
- FAIRCALL – An anonymous and confidential reporting system available to all employees that is used to communicate misconduct and other concerns. The program shall help minimize negative morale, promote a positive work place, and encourage reporting suspected misconduct without retribution. Associated upper management is notified and the investigations are documented.
- Use of flexible work hours. Within reason and as approved by supervisors, employees are allowed flexible work hours in order to help ease schedule pressures which could impact decision-making and work quality.
- Operational and project scheduling assessments are continually made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads. Procedures for subcontracting work are established, and within the ALS Environmental laboratory network additional capacity is typically available for subcontracting, if necessary.
- Gifts and Favors (Code of Conduct Agreement) – To avoid possible conflict of interest implications, employees do not receive unusual gifts or favors to, nor accept such gifts or favors from, persons outside the Company who are, or may be, in any way concerned with the projects on which the Company is professionally engaged.

All employees are required to sign and adhere to the requirements set forth in the *Code of Conduct Agreement, Confidentiality Agreement,* and *Ethics and Data Integrity Agreement*. The *Ethics and Data Integrity Agreement* is signed by all employees on an annual basis (see Appendix C).

3.8 The ALS Environmental-Simi Valley staff, consisting of approximately 30 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds, experience, and provide the comprehensive skills that the laboratory requires. As seasonal workload increases, temporary employees may be hired to perform specific tasks.

ALS Environmental is committed to providing an environment that encourages excellence. All employees share the responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 3-1 lists the ALS Environmental-Simi Valley personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program including ensuring compliance with ISO/IEC 17025:2005 and is responsible for overall laboratory efficiency and the financial performance of the Simi Valley facility.

  The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

- The **Quality Assurance Manager** (QA Manager) has the authority and responsibility for implementing, maintaining, and improving the quality system. This includes coordination of QA activities within the laboratory, ensuring that all personnel understand their contributions to the quality system, ensuring communication takes place at all levels within the laboratory regarding the effectiveness of the quality system, evaluating the effectiveness of training; and monitor trends and continually improve the quality system. Audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews can all be used to support quality system implementation. The QA Manager is responsible for ensuring compliance with all applicable regulatory compliance quality standards (i.e. NELAP/TNI, ISO/IEC 17025:2005, DoD QSM, etc.). The QA Manager works with laboratory staff to establish effective quality control and assessment plans and has the authority to stop work in response to quality problems. The QA Manager is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and ensuring the annual review of technical SOPs; maintaining QA records such as metrological records, archived logbooks, PT results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; and performing internal QA audits.

  The QA Manager reports directly to the Laboratory Director and also reports indirectly to the Manager of Quality Assurance, USA. It is important to note that when evaluating data, the QA Manager does so in an objective manner and free of outside, or managerial, influence.

- The Manager of Quality Assurance, USA is responsible for the overall QA program at all the ALS Environmental Group laboratories. The Manager of Quality Assurance, USA is responsible for oversight of QA Managers regulatory compliance efforts (NELAP/TNI, ISO, DoD, etc) and may perform internal audits to evaluate compliance. The Manager of Quality Assurance, USA approves company-wide SOPs and provides assistance to the laboratory QA staff and laboratory managers as necessary.

➢ In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are a Project Manager or Volatile Organics Technical Manager (for the Laboratory Director) and the Laboratory Director (for the QA Manager).

- In the event that work is stopped in response to quality problems, only the Laboratory Director or QA Manager have the authority to resume work.

- The **Environmental Health and Safety Coordinator** (EH&S) is responsible for the administration of the laboratory health and safety policies.

  This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S Coordinator is also designated as the Chemical Hygiene Officer. The EH&S Coordinator has a dotted-line reporting responsibility to ALS North America EH&S Director.

- The **Data Validation Coordinator/Reporting Supervisor** is responsible for data review, data package preparation, review and coordination, and preparation of case narratives (based on the information provided by the laboratory).

- The **Client Services Manager** is responsible for the Client Services Department defined for the laboratory (i.e. Project Managers, data reporting, etc.) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specifications to final deliverables. Sample management handles all activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.

- The **Project Manager** is a scientist assigned to each client to act as a technical liaison between the client and the laboratory. The Project Manager is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the ALS Environmental laboratory and administrative staff to ensure that client-specific needs are understood and that the services ALS Environmental provides are properly executed and satisfy the requirements of the client.

- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a QC program meeting department needs. Each **Department Manager and Supervisor** has the responsibility to ensure compliance with ISO/IEC 17025:2005, ensure that QC functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.

- The **Sample Management Office** plays a key role in the laboratory QA program by performing and/or assisting in the proper preparation and shipment of sampling media. In addition, personnel are responsible for the verification of sample receipt information, performing sample acceptance and log-in and distribution of documentation per laboratory defined procedures and the initial storage of samples in the proper environment and location and performing proper sample

disposal. Responsibilities also include monitoring and recording of critical thermal preservation equipment temperatures and calibration of associated thermometers against NIST traceable thermometers.

- **Information Technology** (IT) staff is responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.
- The **Procurement Manager** is responsible for directing and coordinating activities of personnel engaged in buying materials and supplies.

Table 3-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Kelly Horiuchi, B.A.	15	Laboratory Director / Project Manager
Chaney Humphrey, B.S.	11	Quality Assurance Manager
Robin Gill	35	Data Validation Coordinator / Reporting Supervisor
Ku-Jih Chen, B.S.	40	Principle Chemist
Sue Anderson, B.S.	25	General (WET) Chemistry Technical Manager / Project Manager
Samantha Henningsen, B.S.	6	Project Manager
Kathleen Aguilera, B.A.	26	Client Services Manager / Project Manager
Wade Henton, B.S.	29	Volatiles (GC) Technical Manager
Chris Parnell, B.S.	29	Operations Manager / Volatiles (GC/MS) Technical Manager
Wida Ang, B.S.,M.S.	30	Volatiles (GC/MS) Team Leader
Madeleine Dangazyan, B.S.	20	Semi-Volatiles / Industrial Hygiene Technical Manager
Jeff Christian, B.S.	36	Director of Operations – Western U.S.

Additional Key Personnel	Project Role
Joe Caulfield	LIMS Manager
Steve Manak	Procurement Group Leader

4) Document Control

4.1 Procedures for control and maintenance of documents are described in the *SOP for Document Control* (CE-GEN005). The requirements of the SOP apply to all laboratory logbooks (standards, maintenance, run logbooks, etc), certificates of analysis, SOPs, QAMs, quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled ALS Environmental documents.

4.2 The contents of this manual are reviewed, revised (as needed) and approved for use at least annually by authorized personnel (QA Manager, Laboratory Director, and Technical Directors) where the scope of the review ensures that it continuously reflects current policies and practices and incorporates all applicable requirements. Additionally, the date the review was completed is indicated by the date of the last approval signature on the title page.

4.3 Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the QA Manager, or designee, and ensures that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file. Logbook entries are standardized following the *SOP for Making Entries onto Analytical Records* (CE-QA007). The entries made into laboratory logbooks are reviewed and approved at a regular interval (quarterly).

4.4 A records system is used which ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data and Record Archiving* (ADM-ARC).

4.5 External documents relative to the management system are managed by the QA Manager. To prevent the use of invalid and/or outdated external documents, the laboratory maintains a master list of current documents and their availability. The list is reviewed before making the documents available. External documents are not issued to personnel.

4.6 Electronic Signatures It is a policy of ALS Environmental to allow the use of electronic signatures. For data reporting an electronic signature may be applied to the report by an approved report signatory and is binding to the same extent as a handwritten wet signature.

To authenticate the electronic signature the identity of the signatory is verified before their electronic signature can be created. Each electronic signature shall be unique to a single individual and shall not be used by any other individual. These signatures are established using only defined procedures within the software and are verified using the two distinct components of *username* and *password*. Each use of the electronic

signature requires entry of the username and the password. The report may not be changed once the signature has been applied.

Additionally, as a form of 'signature' used for LIMS, email, and certain internal documentation processes (e.g. acknowledgements, attestations, audit trails, etc.), and other electronic tools the user's system login credentials are used to verify and authenticate the identity of the user. Following login, these credentials are used to identify and document the user.

5) Review of Requests, Tenders and Contracts

5.1 Procedure for the Review of Work Requests

5.1.1 Requests for new work are reviewed prior to signing any contracts or otherwise agreeing to perform the work. The specific methods to be used are agreed upon between the laboratory and the client. A capability review is performed to determine if the laboratory has or needs to obtain certification to perform the work, to determine if the laboratory has the resources (personnel, equipment, materials, capacity, skills, expertise) to perform the work, and if the laboratory is able to meet the client's required reporting and QC limits. The results of this review are communicated to the client and any potential conflict, deficiency, lack of appropriate accreditation status, or concerns of the ability to complete the client's work are resolved.

5.1.2 Any differences between the request or tender and the contract shall be resolved before any work commences. The client should be notified at this time if work is expected to be subcontracted. Each contract shall be acceptable both to the laboratory and the client. Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work.

5.1.3 If a contract needs to be amended after work has commenced, the contract review process is repeated and any amendments are communicated to all affected personnel. Changes in accreditation status affecting ongoing projects must be reported to the client.

5.2 Allowed Deviations from Standard Operating Procedures

5.2.1 When a client requests a modification to an SOP the Project Manager must discuss the proposed deviation with the laboratory supervisor and obtain approval to accept the project. The Laboratory Director and QA Manager may also be involved. The Project Manager is responsible for documenting the approved or allowed deviation from the SOP.

5.2.2 When a client request necessitates a deviation or departure from company policies or procedure involving any non-technical function, the allowed deviation must be approved by the laboratory or the Laboratory Director. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the Laboratory Director will address the possible need for a change in policy.

6) Subcontracting of Tests

Analytical services are subcontracted when the laboratory needs to balance workload or when the requested analyses are not performed by the laboratory. Subcontracting, to capable qualified laboratories is only done with the knowledge and approval of the client.

Subcontracting to another ALS Environmental laboratory is preferred over external-laboratory subcontracting. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories* (CE-QA004). The QA Manager is responsible for maintaining a list of qualified subcontract laboratories.

7) Purchasing Services and Supplies

The quality level of reagents and materials (grade, traceability, etc.) required is specified in the analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. The *SOP for Handling Consumable Materials* (ADM-CONSUM) provides default expiration requirements. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in the *SOP for Procurement and Control of Laboratory Services and Supplies* (CE-GEN007). Also, refer to section 13.5 for a discussion of reference materials.

Receipt procedures include technical review of the purchase order/request to verify that what was received is identical to the item ordered. The laboratory checks new lots of reagents for unacceptable levels of contamination prior to use in sample preservation, sample preparation, and sample analysis by following the *SOP for Quality of Reagents and Standards* (CE-QA012).

8) Service to the Client

The laboratory uses a number of systems to assess its daily operations. In addition to the routine quality control (QC) measurements, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients including; on-time performance, customer complaints, training reports and non-conformity reports. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

ALS Environmental utilizes a number of different methods to ensure that adequate resources are available for service demands. Senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating sufficient resources. All Requests for Proposal (RFP) documents are reviewed by Project Managers, Business Development and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Project Plans (QAPPs) if available. Status/production meetings are also conducted regularly with the laboratory and project managers to inform the staff of the status of incoming work, future projects, or project requirements.

When a customer requests a modification to an SOP, policy, or standard specification the Project Manager will discuss the proposed deviation with the Laboratory Director and department manager to obtain approval for the deviation. The QA Manager may also be involved. All project-specific requirements must be on-file and with the service request upon logging in the samples. The modification or deviation must be documented. A Project-Specific Communication Form, LIMS comments, or similar, may be used to document such deviations.

The laboratory shall afford clients cooperation to clarify the client's request and to monitor the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients. The laboratory maintains and documents timely communication with the client for the purposes of seeking feedback and clarifying customer requests. Feedback is used and analyzed to improve the quality of services. The *SOP for Handling Customer Feedback* (CE-GEN010) is in place for these events.

9) Complaints

The laboratory maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the Project Manager) is responsible for documenting the complaint. If the Project Manager is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback* (CE-GEN010).

10) Facilities and Equipment

ALS Environmental-Simi Valley maintains approximately 20,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system is designed to meet any needs of analyses performed in the separate work areas. ALS Environmental-Simi Valley minimizes laboratory contamination sources by employing janitorial staff to ensure good housekeeping. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Sample Management Office; Shipping and Receiving
- Records Archival
- Volatile Organics Laboratory (GC and GC/MS)
- Semi-Volatiles Laboratory (GC, GC/MS and HPLC)
- Ultra-Low Level Volatile Organics GC/MS
- General/Wet Chemistry Laboratory
- R&D Laboratory
- Canister Conditioning and Maintenance
- Flow Controller and Critical Orifice Calibration Station
- Sample Storage Walk-in Refrigerator
- Sample, Standards, and Media Storage
- Waste Disposal
- Laboratory Deionized Water System
- Laboratory Management, Client Service, Report Generation and Administration
- Information Technology (IT)

The designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Refer to Appendix D for facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix E lists the major equipment, illustrating the laboratory's overall capabilities and depth.

ALS Environmental-Simi Valley also maintains a satellite extraction facility located at 2360 Shasta Way, Unit G, Simi Valley, California. The approximately 2,000 square foot building contains five fume hoods and is designed with the purpose of performing semi-volatile

organics extraction of air, liquid and solid matrices. The extraction facility is equipped with sufficient bench space, glassware washing equipment and materials, flammable solvent storage, sample/extract storage refrigerators and an electric kiln. Refer to Appendix D for the floor plan of the facility.

10.1 Preventive Maintenance

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at ALS Environmental (e.g., GC/MS systems, gas and liquid chromatographs, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at ALS Environmental contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at ALS Environmental before it may be used for sample analysis. Each instrument must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument or if the continuing calibration verification acceptance criteria have not been met as specified in the standard operating procedure. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at ALS Environmental. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the department supervisor or laboratory director. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the department supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. The laboratory maintains an adequate supply of expendable maintenance items (expected lifetime of part of less than 1 year.) These parts include items needed to perform the preventive maintenance procedures listed in Table 16-1.

When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and

- Demonstration of return to analytical control.

See the Table 16-1 for a list of preventive maintenance activities and frequency for each instrument.

For further information regarding Instrumentation see the *SOP for Analytical Instrument Acquisition, Reassignment, Maintenance and Documentation* (ADM-INSTRUM).

10.2 Temperature Control

Temperatures are monitored and recorded for all critical measurement temperature-regulating devices including freezers, refrigerators and ovens. Each piece of equipment is labeled with a unique identifier, the required temperature or range of use according to the needs of the analysis or application. Temperature record books are kept which contain equipment identifier, daily-recorded temperatures (if in use, business days), acceptance criteria and the initials of the laboratory staff member who performed the checks for all temperature-regulating devices in daily use.

10.3 Water Purification Systems

Purified water is utilized for a number of laboratory functions including instrument and method blanks, trip blanks, washes and sample dilutions. The water purification system utilizes three mixed-ion beds, four filters, and resistively lights with constant water recirculation. It is designed to produce deionized water of ASTM Type II quality, with 16-18 megohm-cm resistance at 25˚C and is checked and recorded daily (prior to and if in use). Maintenance and repair on the system is conducted by an approved service supplier and all records including purification checks/verifications are maintained on file for review. For procedures on additional purification (i.e., boiling and/or purging) and purification checks/verifications, refer to the applicable method standard operating procedures.

11) Sample Management

Standard operating procedures have been established for all aspects of sample management within the laboratory including sample receiving, handling, acceptance, log-in, protection, storage, retention, transportation, and disposal. The procedures include provisions necessary to protect the integrity of the sample (as received) and to protect the interests of the laboratory as well as the client. These procedures ensure that samples are handled properly and that all associated documentation is complete and consistent. The sample handling factors that must be taken into account to ensure accurate, defensible analytical results include but are not limited to:

- Amount of sample taken (sampling)
- Type of container used
- Existence and type of sample preservation
- Holding Time
- Proper custodial documentation
- Sample storage, tracking and/or transfer
- Retention
- Disposal

A record of all procedures to which a sample is subjected while in the possession of the laboratory including acceptance, rejection, login, identification, preservation checks, storage, tracking, and disposal are documented and maintained. In addition, all indirect procedures which support each record of a sample and protects the integrity of a sample is documented and maintained (i.e., refrigerator and freezer temperature checks, thermometer calibrations, etc.).

11.1 Sampling

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples.

ALS Environmental-Simi Valley does not provide sampling services. The laboratory only provides materials needed for sample collection; therefore, ALS Environmental-Simi Valley recommends that clients follow sampling guidelines described in the specific reference methods including 40 CFR 136 and/or USEPA SW-846, NIOSH, OSHA, ASTM, CARB and SCAQMD as appropriate.

When transporting samples to the laboratory, the most expedient but lawful route of transport should be utilized. Also, the hazardous potential of the samples needs to be considered when shipping samples via air freight or passenger airlines.

11.2 Preservation

ALS Environmental-Simi Valley uses sample preservation, container, and holding time recommendations published in a number of referenced documents including, but not limited to USEPA SW 846, USEPA 600/4-79-020, USEPA 600/r-93-100 (inorganic substances), 600/4-91-010, and EPA/625/R-96/010b (air samples) and the US EPA Methods Update Rule effective 4/11/07. The complete citation for each of these and other references can be found in Section 23 of this document. The appropriate container, preservation and holding time information are summarized in Appendix F. Additional information on this is addressed in each corresponding method SOP.

11.3 Shipping of Containers and Samples

ALS Environmental-Simi Valley provides sample containers to clients via media requests for all matrices (soil, water, air) with the appropriate preservatives (as applicable). These containers include Tedlar bags, Summa canisters, silica-gel tubes, etc. ALS Environmental-Simi Valley keeps client-specific shipping requirements on file and utilizes all major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. ALS Environmental-Simi Valley also provides its own courier service that makes scheduled courier runs in the greater Los Angeles metropolitan area. The procedures for all requirements directed toward media requests follow the requirements detailed in the *SOP for Media Request Fulfillment* (ADM-Media_Req).

11.4 Sample Receiving and Acceptance

It is the policy of ALS Environmental-Simi Valley to check and record the condition of each sample (i.e. pressure, temperature, etc.) delivered to the Sample Management Office (SMO) and received by the Sample Management Custodian or alternates against certain acceptance criteria as documented in the *SOP for Sample Receiving, Acceptance, and Log-In* (SMO-SMPL_REC). This policy is available to all sample management personnel for reference. Any samples, which deviate from these outlined areas, will be clearly flagged with the nature and substance of the deviation. Assessment and condition checks utilized by ALS Environmental-Simi Valley for the acceptance or rejection of samples are based on the criteria found in Appendix F, applicable Quality Assurance Project Plan (QAPP), permit, program or rule where appropriate. This verification of sample integrity is conducted by the Sample Custodian and may be dependent on the matrix (i.e., temperature, preservation, and headspace) being submitted.

Any abnormalities or departures from specified condition requirements (as described herein) as observed during the initial assessment are recorded. When there is any doubt as to the suitability of a sample for testing, including signs of damage, when a sample does not conform to the description provided, or when the test method required is not specified in sufficient detail the appropriate Project Manager (PM) is notified.

The Project Manager is to consult with the client, whenever possible, regarding specific integrity issues documented during sample receipt for further instructions before proceeding and retain a written record of discussion. There may be instances where the client is unavailable, in which case the PM shall document all attempts at contacting the client.

There may be a need to inform the client that a sample(s) is rejected and cannot be accepted for analysis into the laboratory. This situation includes, but is not limited to loss of sample or insufficient amount (subsampling may be performed if it would not cause loss of sample integrity, but the procedure must be indicated with the test results). Subsampling as in the case of air samples is not appropriate.

The procedures for sample documentation, handling acceptance requirements and deviations from the sample acceptance policy are discussed in detail in the *SOP for Sample Receiving, Acceptance and Log-In* (SMO-SMPL_REC). This procedure is also in place to ensure samples are received and properly logged into the laboratory, and that all associated sample documentation, including Chain-of-Custody (COC) records are complete and consistent with the samples received. All associated documentation, including chain of custody forms, memos, transmittal forms, and phone logs, are kept with each project file.

11.5 Sample Log-in

Each sample is logged into the laboratory in such a way as to ensure traceability and cross-reference with regards to the unique laboratory job number, sample identifications and client sample identifications. The laboratory identification is retained throughout the life of the sample in the laboratory. The identification system is designed and operated to ensure that samples cannot be confused physically or in laboratory documentation. Additional information is provided in the *SOP for Sample Receiving, Acceptance, and Log-In* (SMO_SMPL_REC).

11.6 Sample Custody

A sample is in someone's "custody" if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked up so that no one can tamper with it;
4. It is kept in a secured area, restricted to authorized personnel only.

Chain-of-Custody (COC) records are used to establish the legal custody of samples, showing the continuous possession of samples from sample collection and transportation to final destination at the laboratory. Custody of each sample is maintained from receipt through disposal (internally utilizing LIMS). When environmental samples are shipped to other laboratories for analysis, the sample management office follows formalized procedures for maintaining the chain of custody, which is written in SOPs for Sample Receiving, Acceptance and Login and Laboratory Storage, Analysis, and Tracking.

When samples are removed from the fixed lab and transported to the off-site extraction facility for sample preparation, internal chain of custody procedures still apply. When sample preparation is completed, sample extracts are returned to the laboratory.

Laboratory security and access is important in maintaining the integrity of samples received at ALS Environmental-Simi Valley.

Access to the building is limited to the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area.

The laboratory is equipped with an alarm system which is monitored by a private security firm who provides nighttime and weekend security.

11.7 Sample Storage, Analysis and Tracking

The procedures and requirements for documenting the storage, analysis and tracking as well as maintaining integrity of samples are detailed in the *SOP for Laboratory Storage, Analysis, and Tracking* (ADM-LabSAT).

11.8 Sample Retention and Waste Disposal

Upon completion of all analyses, the laboratory samples are retained in accordance with the requirements specified in the method SOPs and the *SOP for Waste Disposal* (ADM-Waste). The samples are disposed according to approved disposal practices or returned to the client (if applicable). All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. This evaluation is generally based on results from analyses performed on the sample by ALS Environmental-Simi Valley or an approved subcontract laboratory. It should be noted that all wastes produced at the laboratory, including the laboratory's own various hazardous waste streams, are treated in accordance with all applicable local, State and Federal laws. Complete documentation is maintained for samples from initial receipt through final disposal. This ensures an accurate record of the samples from "cradle to grave."

11.9 Intra-laboratory / Inter-laboratory Transfer of Samples

When environmental samples are shipped to another laboratory for analysis, samples are properly packed for shipment and preserved if necessary. Sample bottles are wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during the transportation process. Blue or wet ice is used for temperature preservation, where necessary.

Figure 11-1
Air Chain of Custody Form

Air - Chain of Custody Record & Analytical Service Request

Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

Company Name & Address (Reporting Information)

Project Name

Project Number

ALS Contact:

Analysis Method

Project Manager

P.O. # / Billing Information

Phone | Fax

Email Address for Result Reporting

Sampler (Print & Sign)

Comments
e.g. Actual Preservative or specific instructions

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Canister ID (Bar code # - AC, SC, etc.)	Flow Controller ID (Bar code #- FC #)	Canister Start Pressure "Hg	Canister End Pressure "Hg/psig	Sample Volume			

Report Tier Levels - please select

Tier I - Results (Default if not specified) _____ Tier III (Results + QC & Calibration Summaries) _____
Tier II (Results + QC Summaries) _____ Tier IV (Data Validation Package) 10% Surcharge _____

EDD required Yes / No
Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:	
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:	Cooler / Blank Temperature ____°C

Figure 11-2
Soil / Water Chain of Custody Form

Soil / Water - Chain of Custody Record & Analytical Service Request Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

ALS Contact:

Company Name & Address (Reporting Information)

Project Name

Project Number

Project Manager

P.O. # / Credit Card / Billing Information

Phone | Fax

Email Address for Result Reporting

Analysis

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Water	Soil	Solid	Other				Comments

Report Tier Levels - please select
Tier I - Results (Default if not specified) ____ Tier III (Results + QC & Calibration Summaries) ____ EDD required Yes / No
Tier II (Results + QC Summaries) ____ Tier IV (Data Validation Package) 10% Surcharge ____ Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs)

Reliquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Reliquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Reliquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:

Cooler / Blank / Ice / No Ice
Temperature _____ °C

Figure 11-3

ALS Environmental
Sample Acceptance Check Form

Client: _____ Work order: _____
Project: _____
Sample(s) received on: _____ Date opened: _____ by: _____

Note: This form is used for all samples received by ALS. The use of this form for custody seals is strictly meant to indicate presence/absence and not as an indication of compliance or nonconformity. Thermal preservation and pH will only be evaluated either at the request of the client and/or as required by the method/SOP.

		Yes	No	N/A
1	Were **sample containers** properly marked with client sample ID?	☐	☐	☐
2	Container(s) **supplied by ALS**?	☐	☐	☐
3	Did **sample containers** arrive in good condition?	☐	☐	☐
4	Were **chain-of-custody** papers used and filled out?	☐	☐	☐
5	Did **sample container labels** and/or tags agree with custody papers?	☐	☐	☐
6	Was **sample volume** received adequate for analysis?	☐	☐	☐
7	Are samples within specified holding times?	☐	☐	☐
8	Was proper **temperature** (thermal preservation) of cooler at receipt adhered to?	☐	☐	☐
9	Was a **trip blank** received?	☐	☐	☐
10	Were **custody seals** on outside of cooler/Box?	☐	☐	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☐
	Were signature and date included?	☐	☐	☐
	Were seals intact?	☐	☐	☐
	Were custody seals on outside of sample container?	☐	☐	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☐
	Were signature and date included?	☐	☐	☐
	Were seals intact?	☐	☐	☐
11	Do containers have appropriate **preservation**, according to method/SOP or Client specified information?	☐	☐	☐
	Is there a client indication that the submitted samples are **pH** preserved?	☐	☐	☐
	Were **VOA vials** checked for presence/absence of air bubbles?	☐	☐	☐
	Does the client/method/SOP require that the analyst check the sample pH and if necessary alter it?	☐	☐	☐
12	**Tubes:** Are the tubes capped and intact?	☐	☐	☐
	Do they contain moisture?	☐	☐	☐
13	**Badges:** Are the badges properly capped and intact?	☐	☐	☐
	Are dual bed badges separated and individually capped and intact?	☐	☐	☐

Lab Sample ID	Container Description	Required pH *	Received pH	Adjusted pH	VOA Headspace (Presence/Absence)	Receipt / Preservation Comments

Explain any discrepancies: (include lab sample ID numbers): _____

RSK - MEEPP, HCL (pH<2); RSK - CO2, (pH 5-8); Sulfur (pH>4)

12) Analytical Procedures

ALS Environmental employs methods and analytical procedures from a variety of external sources. Reference documents include but are not limited to: ASTM, CARB, NCASI, NIOSH, OSHA, SCAQMD, USEPA SW-846, USEPA 600/4-79-020, 600/4-91-010, 600/R-93/100 (inorganic substances), 600/625/R-96/010b (air samples), EPA 40 CFR part 136 and associated Method Update Rules and Supplements, and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 23. Other published procedures, such as state-specific methods, program-specific methods, or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by ALS Environmental is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix J. Further details are described below.

12.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks

ALS Environmental maintains SOPs for use in both technical and administrative functions (Refer to Appendix G). SOPs are written following standardized format and content requirements as described in the *SOP for Establishing Standard Operating Procedures* (CE-GEN009). Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the QA Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents, unless otherwise noted. The procedures for document control are described in the *SOP for Document Control* (CE-GEN005). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries onto Analytical Records* (CE-QA007). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

12.2 Modified Procedures

ALS Environmental strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

12.3 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that ALS Environmental-Simi Valley has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.
2) All (field) samples in a batch are of the same matrix.
3) The QC samples to be processed with the (field) samples include:
   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination

   b) Laboratory Control Sample

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.
4) A single lot of reagents is used to process the batch of samples.
5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.
6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours between the start of processing of the first and last sample of the batch.
7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.
8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).
9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.
10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.
11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

Note: Matrix spiked samples are often not feasible for air matrices. Therefore, the MS shall be used as required by the test method and as specified by the corresponding method SOP.

12.4 Specialized Procedures

ALS Environmental not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are specialized GC/MS analyses, and low level organics analyses (including PAHs, pesticides and PCBs).

12.5 Demonstration of Capability

A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria are met.

12.6 Method Detection Limits and Method Reporting Limits & Limits of Detection/ Quantitation

Method Detection Limits (MDL) for methods performed at ALS Environmental-Simi Valley are determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation* (CE-QA011), which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. limit of quantitation - LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

13) Measurement Traceability and Calibration

All equipment and instruments used at ALS Environmental are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files. Records are maintained to provide traceability of reference materials.

Traceability is defined as the property of a measurement result or value of a standard which can be related to stated references through an unbroken chain, each with stated uncertainties and is documented for all material used to perform calibrations. The documentation, a certificate of analysis containing, at a minimum, the manufacturer, address, accreditation number (where applicable), how traceability was achieved, the traceable values, their associated uncertainty, and the unique serial or laboratory identification number of the equipment or standard reference material (SRM) shall serve as initial point in the chain of traceability. The unique serial number or laboratory identification number is used throughout the laboratory to trace equipment and materials back to the original certificate of analysis.

Laboratory support equipment (thermometers, balances, and weights) are verified on an annual basis by a vendor accredited to ISO/IEC 17025:2005 International Standards. All analytical measurements generated at ALS Environmental are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

Equipment subjected to overloading or mishandling, or has been shown by verification to be defective, is taken out of service and labeled until repaired. That piece of equipment is placed back in service only after verifying, by calibration, that it performs satisfactorily.

13.1 Temperature Measuring Devices

All thermometers are identified by a unique identifying number (i.e., serial number), and the calibration of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. All corresponding correction factors are noted on the device as well as in the thermometer calibration logbook. The NIST calibrated thermometer is recertified by an approved vendor accredited ISO/IEC 17025:2005 International Standard on an annual basis and certificates are retained on file for review. All temperature monitoring is conducted in accordance with the *SOP for Sample Receipt, Acceptance and Log-In* (SMO-SMPL_REC) and thermometer calibration requirements are performed in accordance with the *SOP for Calibration and Use of the Laboratory Support Equipment* (ADM-SupEQ).

A number of thermometers include a temperature range per certain project requirements (complies with Department of Defense Quality Systems Manual for Environmental Laboratories); this range is recorded to document consistent compliance with required temperatures for refrigerators and freezers, where applicable.

13.2 Volumetric Dispensing Devices

The accuracy of pipettes used to make critical-volume measurements is verified on a quarterly basis. Typically, the indicated volume or range (where applicable) of the pipette is checked and both the accuracy and precision verification are performed using the above-mentioned procedure. The calibrations are evaluated against the intended use (volume or range) of the pipette and if the calibration is not approved for the specified volume(s) it is tagged accordingly (i.e. "Do Not Use Below 5uL"). The results for all calibration verifications are recorded and maintained.

Note: Glass microliter syringes including gas-tight syringes are considered in the same manner as Class A glassware and are not held to the calibration/verification requirements as are other volumetric dispensing devices.

13.3 Analytical Balances and Weights

Analytical balances and weights are calibrated/recertified and certificates issued annually by an approved vendor accredited to ISO/IEC 17025:2005 International Standard. The calibration of each balance is checked once each day of use in the expected range, utilizing the calibrated weights. Bound record books are kept which contain the identification of balance (serial number), recorded measurements and the initials of the analyst who performed the check. All certificates for the balances and weights are available for review.

13.4 Pressure/Vacuum Gauges

ALS Environmental-Simi Valley digital pressure/vacuum gauges are used in a number of critical measurements within the laboratory. The following is a list of the uses for this gauge type.

- Canister cleaning and conditioning
- Measure the vacuum on canisters before they are sent to the client for sampling.
- Measure the initial/final vacuum/pressure of canisters prior to analysis.
- Measure pressure during the preparation of selected standards.

Digital pressure/vacuum gauges are calibrated and certificates issued once per year by an approved metrology organization. All calibrations are performed against standards traceable to the National Institute of Standards and Technology (NIST) or other recognized national metrology institutes. In addition, ALS Environmental-Simi Valley performs a calibration check for each gauge six months following the calibration date. The laboratory retains all corresponding calibration and verification documentation for review.

13.5 Source and Preparation of Standards and Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors where possible have fulfilled the requirements for ISO 9001 certification and/or are ISO 17025 accredited. ALS Environmental-Simi Valley relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination.

The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries onto Analytical Records* (CE-QA007). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material.

13.6 Instrument Calibration

The laboratory specifies the procedures and documentation for initial instrument calibration and continuing calibration verification in the applicable method standard operating procedures to ensure that data is of known quality and is appropriate for a specific regulation and/or client requirement. The procedural steps for calibration including, frequency, number of points, integration, calculations, acceptance criteria (appropriate to the calibration technique employed), corrective action, associated statistics, and data qualifications are included in applicable methods, method standard operating procedures and/or client project plans. The essential elements that define the procedures and required documentation for initial instrument calibrations are specified below.

- Sufficient raw data records are retained to permit reconstruction of all calibrations.
- If a reference or mandated method does not specify the number of calibration standards, the initial calibration range shall consist of a minimum of 5 contiguous calibration points for organics and a minimum of 3 contiguous calibration points for inorganics. The actual numbers of points utilized is specified in the corresponding method SOP.
- The concentrations should bracket the expected concentration range of samples.
- Initial instrument calibration procedures referenced in test methods (either directly or indirectly) are readily available to the analysts.
- All sample results are quantitated from the initial instrument calibration and are not quantitated from any continuing instrument calibration verification unless otherwise specified by regulation, method or program.
- The initial instrument calibration is verified with a standard obtained from a second manufacturer or lot and traceability to a national standard is maintained, where available.
- The acceptance criteria utilized is appropriate for the calibration technique employed.
- The lowest calibration standard in the initial calibration is at or below the lowest concentration for which quantitative data are to be reported and is referred to at this laboratory as the method reporting limit (MRL). Some programs and/or agencies refer to this limit as the practical quantitation limit (PQL) or Limit of Quantitation (LOQ).
- Any data reported below the MRL or above the highest calibration standard is considered to have an increased quantitative uncertainty and is appropriately qualified in the report.

- The lowest calibration standard is above the limit of detection or method detection limit (MDL).

13.7 Internal and External Calibrations

Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area of the target compound in the sample or sample extract to the peak area of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF) or relative response factor (RRF) in some methods.

External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector responses to the amount (mass) of analyte in the calibration standard is defined as the calibration factor or in some cases it may be referred to as response factor.

13.8 Continuing Calibration Verification

The essential elements that define the procedures and required documentation for continuing instrument calibration verification are specified below.

- When an initial calibration is not performed on the day of analysis, continuing instrument calibration verification is analyzed with each batch.
- Calibration is verified for each reported compound, element or parameter; however, for multi-component analytes such as aroclors or total petroleum hydrocarbons a representative chemical related substance or mixture may be used. The allowance for this exception is dependent on applicable regulatory, method, or client project plans.
- Generally, the instrument calibration verification is performed at the beginning, end, and every ten samples of each analytical batch (except, if an internal standard is used, only one verification needs to be performed at the beginning of the analytical batch); whenever it is suspected that the analytical system may be out of calibration; if the time period for calibration or most previous calibration verification has expired; or for analytical systems that contain a specific calibration verification requirement. Specific requirements for the frequency of continuing calibration verification, for a particular method, is specified in the corresponding method standard operating procedure.

14) Assuring the Quality of Results

A primary focus of ALS Environmental's QA Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. ALS Environmental has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

14.1 Quality Control Objectives

14.1.1 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination).

ALS Environmental utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement.

14.1.2 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

14.1.3 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the QA Manager. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

14.1.4 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. ALS Environmental has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample. Air samples received by the laboratory in canisters and bags are considered to be homogenous and therefore, no special sample preparation procedures are necessary.

14.1.5 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using ALS Environmental or project-specified data qualifiers.

14.2 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below.

14.2.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (air, water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

14.2.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

14.2.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed.

The frequency of CCB analysis is once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

14.2.4 Calibration Standards

Calibration standards are vapors, liquids or solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

14.2.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used for calibration. Once it is determined that there is no defect or error in the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

14.2.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

14.2.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

14.2.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

14.2.9 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free liquid, solid or air matrix to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

14.2.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water samples.

14.2.11 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

Recovery (%) = (S - A) x 100 ÷ T

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

Note: Matrix spiked samples are often not feasible for air matrices. Therefore, the MS shall be used as required by the test method and as specified by the corresponding method SOP.

14.2.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample.

Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$\textit{Relative Percent Difference (RPD)} = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

14.2.13 Control Charting

The generation of control charts is routinely performed at ALS Environmental. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Limits* (CE-QA009).

14.2.14 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at ALS Environmental undergoes a rigorous cleansing procedure prior to every usage. The *SOP for Glassware Cleaning* (ADM-GLASS) has been generated and outlines the various procedures used at ALS Environmental-Simi Valley; each procedure is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

14.2.15 Collection Efficiency

In the case of sampling trains (consisting of one or more multi-section sorbent tubes), which are received intact by the laboratory, the “front” and “back” sections shall be separated if required by the client. Each section shall be processed and analyzed separately and the analytical results reported accordingly.

14.2.16 Desorption Efficiency and Method Reporting Limits (Industrial Hygiene)

Desorption efficiency (DE) is the ability of an analytical method to recover the analyte from the collection media. Desorption efficiencies are determined initially and for each analyte to be reported. In addition, a DE study is performed each time there is a change in the test method, or with each new lot of media. Desorption efficiency shall be determined using sorbent media from the same lot number used for the field samples, if possible, and of the identical size and type. The DE values are used to correct the sample results (for all samples except passive samplers) before reporting.

Minimum reporting limits for each reportable analyte are determined initially by the analysis of spiked media, prepared at the desired reporting limit and carried through the entire analytical process. The reporting limit is verified or re-established annually (or if there is a change in methodology or instrumentation) and instrument performance is checked with each analytical batch through the analysis of an analytical standard prepared at the reporting limit.

14.2.17 Field and Trip Blanks

Field and trip blanks are analyzed when they are submitted to the laboratory for analysis. The actual field samples are flagged (when analytes are found in the blank) if and only if the laboratory is able to analyze the samples in the same analytical sequence as the corresponding field or trip blank. If this is not possible due to client submission restrictions then the results for the samples and blanks shall be reported independently with no flag. However, an explanation of this is included in the final report. This laboratory does not feel that Summa canisters are suitable for use as trip blanks. It is for this reason that the results for these types of containers are reported as separate samples and flagging is not considered appropriate.

14.3 Uncertainty

When requested by the client or relevant to the validity of reported results, the estimation of measurement uncertainty will be provided to a client or regulatory agency. How the uncertainty will be reported may be dictated by the client's reporting specifications. Procedures for determining and reporting uncertainty are given in the *SOP for Estimation of Uncertainty of Analytical Measurements* (CE-QA010).

15) Control of Non-Conforming Environmental Testing Work

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s) (See Appendix H). Failure to meet established analytical controls, such as the quality control objectives, prompts corrective action. Corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, department manager, and/or the QA Manager may examine and pursue alternative solutions. In addition, the appropriate Project Manager is notified in order to ascertain if the client needs to be notified.

16) Corrective Action, Preventive Action, and Improvement

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using a *Nonconformity and Corrective Action Report* form. The procedure and responsibilities for addressing nonconforming work is defined in the *SOP for Nonconformance and Corrective Action* (CE-QA008). Nonconformances are reported to the client using various means (voice, email, narrative, etc). When a nonconformance occurs that casts doubt on the validity of the test results or additional client instructions are needed, the Project Manager notifies the client the same business day that the nonconformance is confirmed and reported. The QA Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the QA Manager. The QA Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate Project Manager is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

Part of the corrective action process involves determining the root cause. Identifying the root cause of a nonconformance can be difficult, but important for implementing effective corrective action. Root cause principles are used to determine assignable causes, which leads to corrective action taken to prevent recurrence.

16.1 Preventive Action and Improvement

Various preventive action and improvement processes are used for eliminating potential problems or averting problems before they occur. This is explained in the *SOP for Preventive Action* (CE-GEN004).

Table 16-1

Equipment Maintenance Procedures

Instrument	Applicable Activity	Frequency	Performed
Gas Chromatographs	Replace septum	As required	In-House and Outside Vendor
	Check system for gas leaks, loose/fray wires and insulation	With cylinder change/Open system	
	Replace injection port liner	As required	
	ECD wipe test	Every 6 months	
	Thermally Clean ECD	As needed	
	Clean FID	As required	
	Change TCD assembly	As required	
	SCD – Change reaction tube	As required	
	Catalyst check	As required	
Gas Chromatography / Mass Spectrometers	Tune MSD	As needed	In-House and Outside Vendor
	Change Semi-VOA capillary column	As needed	
	Change Semi-VOA injection port septum	As required	
	Change Semi-VOA injection port liner	As required	
	Replace trap (VOA)	As required	
	Clean ion source	As required	
	Change filament	As required	
	Change electron multiplier	As required	
	Vacuum System: • Mechanical pumps: change oil, change trap pellets (HP only) • Diffusion pump: check oil, check cooling fan, change oil • Turbo pump	• Check every 6 months, check level monthly, change at least annually or sooner is necessary • As required • Replace as required	In-House
	Air Preconcentrators / Autosampler: • Change traps • Inspect Rotors • Calibrate Mass Flow Controllers	• As required • As required • Every 6 months	In-House

Instrument	Applicable Activity	Frequency	Performed
HPLC	Replace/clean check valve filter	As required	In-House
	Replace lamp UV/vis detector	As required	
	Replace flow cell	As required	
	Check flow	Quarterly	
Analytical Balances	Clean pan and compartment	Prior to and after use	In-House and Outside Vendor
	Check with NIST traceable weights	Prior to use	
	Field service	Annually	
Refrigerators and Freezers	Monitor Temperature	Daily	In-House
	Adjust Temperature	As required	
	Clean, Defrost	As required	
Ovens	Clean	As needed or if temperature is outside limit	In-House
pH probes	Condition probe	When fluctuations occur	In-House
	Change Filling Solution	Weekly	
Fluoride ISE	Store in storage solution	Between uses	In-House
Ammonia ISE	Store in storage solution	Between uses	In-House
UV-visible Spectrophotometer	Wavelength check	Annually	In-House
Ion Chromatographs	Change column bed supports	Monthly or as needed	In-House
	Clean column	Monthly or as needed	
	Change column	Every six months or as needed	
	Change valve port face and hex nut	Every six months or as needed	
	Clean valve slider	Every six months or as needed	
	Change tubing	Annually or as needed	
	Eluent pump	Annually	
Restek Thermal Gas Purifier	Check getter tube	Monthly, change as required	In-House

17) Control of Records

17.1 Documentation

ALS Environmental maintains a records system which ensures that all laboratory records of analysis data are retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. Archival procedures are described in the *SOP for Data and Record Archiving* (ADM-ARC).

17.1.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes, but is not limited to, the following items (where applicable) for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes, duplicates and reruns;
- Applicable standard identification numbers;
- Chain of custody, service request and sample acceptance check forms;
- Initial calibration and data review checklist(s);
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

17.2 Information Technology

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. ALS Environmental management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies.

17.2.1 Software Quality Assurance

Practices are defined for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the *SOP for Software and Data Quality Assurance* (ADM-SftwreQA).

The purpose of the SOP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of this plan are policies for software validation and control.

17.2.2 IT Support

The local ALS Environmental Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data described in the *SOP for Electronic Data Backup, Archiving, and Restoration* (ADM-DATA_BU). A software inventory is maintained. Additional IT responsibilities are described in the *SOP for Software and Data Quality Assurance* (ADM-SftwreQA).

In addition to the local IT department, ALS Environmental corporate IT provides support for network-wide systems. ALS Environmental also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

17.2.3 Information Management Systems

ALS Environmental has various systems in place to address specific data management needs. The Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system defines sample identification, analysis specifications, and provides a means of sample tracking. This system is used during sample login to generate the internal service request.

Included on the service request is a summary of client information, sample identification, required analyses, work instructions, and deliverable requirements. The LIMS is used to track the status of a sample and is important in maintaining internal chain of custody.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows Server 2008 R2). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats.

17.2.4 Backup and Security

Laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving.

Full backups onto a hard drive are performed on all file server information once per day. In addition, the laboratory's data warehouse located in Canada performs an offsite full backup nightly.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non-ALS Environmental access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. ALS Environmental uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent or comparable service. The external messaging system operates through a single secure gateway. E-mail attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems with Internet access.

18) Audits

Quality audits are an essential part of ALS Environmental-Simi Valley's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

18.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of ALS Environmental-Simi Valley are conducted regularly by various regulatory agencies and clients. Appendix J lists the certification and accreditation programs in which ALS Environmental-Simi Valley participates. Programs and certifications are added as required. Additionally, internal system audits of ALS Environmental-Simi Valley are conducted regularly under the direction of the QA Manager. The internal audit procedures are described in the *SOP for Internal Audits* (CE-QA001). The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that all elements of the ALS Quality System are assessed.
- Technical/method audits
- Hardcopy report audits

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

18.2 Performance Audits

ALS Environmental-Simi Valley also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. General procedures for these analyses are described in the *SOP for Proficiency Sample Testing Analysis* (CE-QA006). ALS Environmental-Simi Valley routinely participates in the following studies:

- American Industrial Hygiene Association (AIHA) PT Program, 4 per year
- Air and Emissions PT studies, 2 per year
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the QA Manager, Laboratory Director, the laboratory staff, and the Manager of Quality Assurance, USA. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformance (NCAR) procedures.

19) Management Review

Quality assurance requires an active, ongoing commitment by ALS Environmental personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) may also be initiated. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed.

It is the responsibility of each laboratory unit to provide the reporting department with reviewed data accompanied by signature approval. The data validation coordinators provide the Project Manager with a final report of the data. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate Project Manager, who in turn reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative is written (or approved) by the Project Manager to explain any unusual problems with a specific analysis or sample, etc.

The QA Manager provides overview support to the Project Managers as required (e.g., contractually specified, etc.). The QA Manager is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QA Manager regularly communicates with the Laboratory Director to review the various QA/QC activities, priorities, and status of program implementation; including such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

An annual management review of the quality and testing systems is performed as described in the *SOP for Laboratory Management Review* (CE-QA005). This is done to identify any necessary changes or improvements to the quality system or quality assurance policies. This review is documented in a Managerial Review of the Laboratory's Quality Systems and Testing Activities and sent to senior management.

20) Personnel

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at ALS Environmental are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at ALS Environmental when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at ALS Environmental. Safety training begins with reading the *Environmental Health and Safety Manual.* Employees are also required to participate in periodic safety training performed by the Environmental, Health and Safety Coordinator.

Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s). Quality Systems training begins with Quality Assurance orientation for new employees and reading the Quality Assurance Manual. During the employee's first month, the employee receives Ethics training and learns about ALS Environmental quality systems. Each employee participates in annual Ethics Refresher training.

ALS Environmental also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The corporate, company-wide training and development program, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained by the QA department. Training requirements and its documentation are described in the *SOP for Training Policy* (CE-QA003). A training plan is developed whenever an employee starts a new procedure or new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

20.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.
- Where one of the three above is not possible training is performed and supervisor approval is documented.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 20-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

20.2 Continuing Demonstration of Proficiency

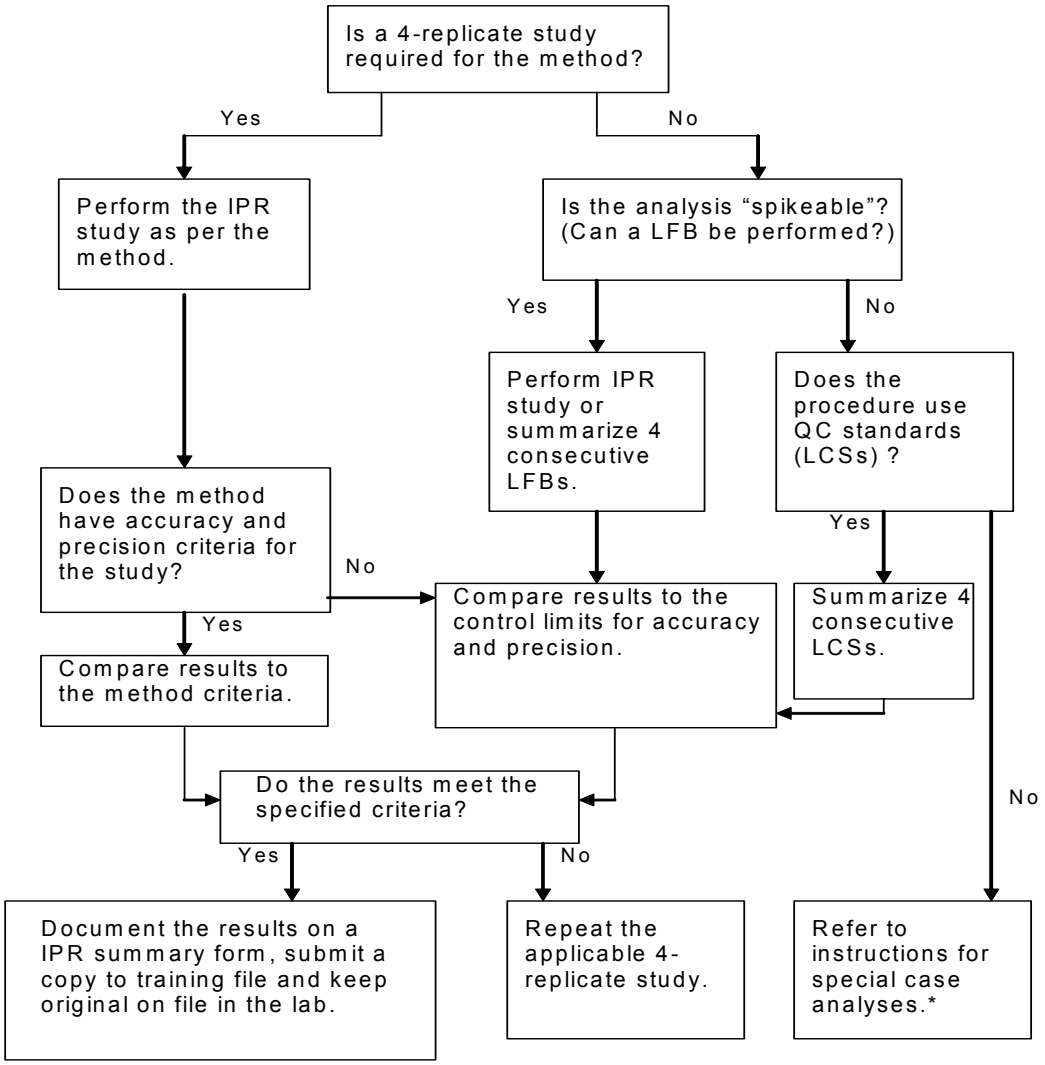
A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

20.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and ALS Environmental resumes. QA maintains a database to record the various technical skills and training acquired while employed by ALS Environmental. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Training Policy* (CE-QA003).

Figure 20-1
Initial Demonstration of Capability Requirements[a]



[a] For IDOC IPR or LFB studies, "second-source" reference materials are used, as per TNI/NELAP requirements
* Refer to the SOP for Training Policy for details. References for Quality Systems, External Documents, Manuals, Standards, and Analytical Procedures

21) Reporting of Results

ALS Environmental reports the analytical data produced in its laboratories to the client via the certified analytical report. This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

21.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). The data is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the supervisor or second qualified analyst reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable it is turned into the reporting department where final reports are generated and then validated by a Data Validation Coordinator. The hardcopy or electronic final report is physically or electronically signed by the project manager and the final report may be stored electronically or in hardcopy format. Test analysis data shall be kept in the appropriate service request folder. Data review and reporting procedures are described in the *SOP for Data Review and Reporting* (ADM-DATA_REV).

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries onto Analytical Records* (CE-QA007).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the "before" and "after" integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration Policy* (CE-QA002).

21.2 Confirmation Analysis

21.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed as required by the method, typically by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.
- The sample meets all of the following requirements:
    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring.

Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.

2. All analytes have been previously analyzed in sample(s) from the same source, identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

21.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
  1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).
  2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS Methods - Two criteria are used to verify identification:
  1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
  2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

21.3 Data Review and Validation of Results

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any

problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.

- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits.

  The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly.
- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. When dilutions are performed the MRL is elevated accordingly.
- Surrogate Results (Organic) – The percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present.

  If no matrix interferences are present and there is no cause for the outlier, the sample is reanalyzed. However, if the recovery is above the upper control limit

with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.

- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used and the results are compared to the MRL. The samples and duplicates are reanalyzed and if re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples reanalyzed or the data reported with the appropriate qualifiers.
- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

21.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package will undergo a peer review by a trained chemist. Prior to release of the report to the client, the Project Manager reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw test data, along with a copy of the final report, is retained by service request number for archival purposes. ALS Environmental maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data is calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Review and Reporting* (ADM-DATA_REV) addresses the flagging and qualification of data. The ALS Environmental-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the analyst or project manager to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Manager verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were

performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Manager accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the ALS Environmental client.

21.5 Deliverables

In order to meet individual project needs, ALS Environmental provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 21-1. Variations may be provided based on client or project specifications.

When requested, ALS Environmental provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. ALS Environmental is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the final report for accuracy.

Table 21-1
Descriptions of ALS Environmental Standard Data Deliverables

Tier I. Routine Certified Analytical Report includes the following:

1. Transmittal letter
2. Chain of custody documents and sample/cooler receipt documentation
3. Sample analytical results
4. Method blank results
5. Surrogate recovery results and acceptance criteria for applicable organic methods
6. Dates of sample preparation and analysis for all tests
7. Case narrative - optional

Tier II. In addition to the Tier I Deliverables, this includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Laboratory Control Sample result(s) with calculated recovery and including associated acceptance criteria
4. Case narrative - optional

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this includes the following:

1. Case narrative - required
2. Summary forms for all associated QC and Calibration parameters, with associated control criteria/acceptance limits

Note: Other summary forms specified in QAPPs or project/program protocols, or those related to specialized analyses such as HRGC/MS will be included.

Tier IV. Full Data Validation Package:

1. All raw data associated with the sample analysis, including but not limited to:
   a. Preparation and analysis bench sheets and instrument printouts,
   b. For organics analyses, all applicable chromatograms, spectral, confirmation, and manual integration raw data. For GC/MS this includes tuning results, mass spectra of all positive hits, and the results and spectra of TIC compounds when requested.
   c. QC data,
   d. Calibration data (initial, verification, continuing, etc),
   e. Calibration blanks or instrument blanks (as appropriate to method).
2. If a project QAPP or program protocol applies, the report will be presented as required by the QAPP.

22) Summary of Changes and Document History

Revision Number	Effective Date	Document Editor	Description of Changes
29	05/30/15	C. Humphrey	2.1 – Revised and added company website
			3.1 – Revised to update NELAC references to NELAP
			3.2 – Revised to update NELAC references to NELAP
			3.8 – Revised to update NELAC references to NELAP
			3.8 – Added Procurement Manager
			Table 3-1 – Added LIMS Manager and Procurement Manager. Updated years of experience.
			12 – Revised to be inclusive of all Method Update Rules and Supplements
			21.1 – Reworded to include both electronic and hardcopy data reduction and review procedures.
			23 – Updated AIHA Policy Modules reference Appendix A – Removed 'NELAC' from glossary
			Appendix B – Updated organization charts
			Appendix E – Updated
			Appendix G – Updated
			Appendix I – Updated
			Appendix J - Updated

23) References for Quality System Standards, External Documents, Manuals, and Test Procedures

The analytical methods used at ALS Environmental generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at ALS Environmental are taken from the references listed below. Additional QA program documents are listed in Appendix I.

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- 2009 TNI Standards.
- American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E).
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 4.2, 10/25/2010.
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 5.0, July 2013.
- American Industrial Hygiene Association-LAP, LLC Policy Document Modules (2A Revision 14, Effective July 1, 2015; 2B Revision 13, Effective July 1, 2015; 6 Revision 3, Effective July 1, 2015), Appendix G (Revision 4, Effective July 1, 2015), and Appendix H (Revision 3, Effective July 1, 2015).
- 3M Organic Vapor Monitor Sampling and Analysis Guide, *Organic Vapor Monitors 3500/3510 and Organic Vapor Monitors 3520/3530*, Technical Bulletin 1028, January 1, 2004.
- 40 CFR Part 60, Test Methods for Standards of Performance for New Stationary Sources, Appendix A.

- 40 CFR Part 63, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Appendix A.
- 40 CFR Part 63, National Emission Standards for Hazardous Air Pollutants for Source Categories, Subchapter C.
- 40 CFR Part 136, Definition and Procedure for the Determination of the Method Detection Limit, Appendix B
- American Society for Testing and Materials (ASTM), Gaseous Fuel, Coal and Coke, Volume 05.06, September 2006.
- American Society for Testing and Materials (ASTM), Annual Book of ASTM Standards, Philadelphia, PA.
- Arizona Administrative Code, *Department of Health Services – Laboratories*, Title 9, Ch. 14, Article 6. *Licensing of Environmental Laboratories*, R9-14-601 through R9-14-621, December 31, 2006 (Supp. 06-4)
- California Environmental Protection Agency Air Resources Board, *Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources*, Volume 3, July 28, 1997.
- California Code of Regulations (CCR), Title 22, Chapter 11 *Identification and Listing of Hazardous Waste*, 7/20/05.
- Minnesota Administrative Rules, *Department of Health*, Chapter 4740, Laboratories; Accreditation Requirements.
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- Environmental Protection Agency, Methods Update Rule (MUR), Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40 CFR Parts 122, 136, 143, 430, 455 & 465; Final Rule 3/12/07, Effective April 11, 2007.
- Environmental Protection Agency, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, Third Edition, 1986 and Updates I (7/92), II (9/94), III (12/96), IIIA (4/98), IIIB (11/04), IVA & IVB. See Chapters 1, 2, 3, 4, 5, 6, and 8.
- Environmental Protection Agency, *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, 1983.
- Environmental Protection Agency, *Methods for the Determination of Inorganic Substances in Environmental Samples,* EPA 600/R-93-100, August 1993.
- Environmental Protection Agency, *EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, Second Edition, EPA/625/R-96-010b, January 1999.
- Environmental Protection Agency, *EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, Second Edition Addendum, October 4, 2000.
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994); 1st Supplement Publication 96-135, 2nd Supplement Publication 98-119, 3rd Supplement 2003-154
- National Council for Air and Stream Improvement, Inc. (NCASI). 2007. *Appendix E - Technical Bulletin Cross Reference Guide for NCASI Methods.* Methods Manual (05).

- *SKC 575 Series Passive Sampler Rate/Selection Guide,* Form #37021, Rev 0012.
- *Standard Methods for the Examination of Water and Wastewater*, 20th Edition (1998).
- South Coast Air Quality Management District, *Laboratory Methods of Analysis for Enforcement Samples.*
- U.S. Department of Labor, Occupational Safety and Health Administration *OSHA Analytical Methods Manual.*

APPENDIX A – Glossary

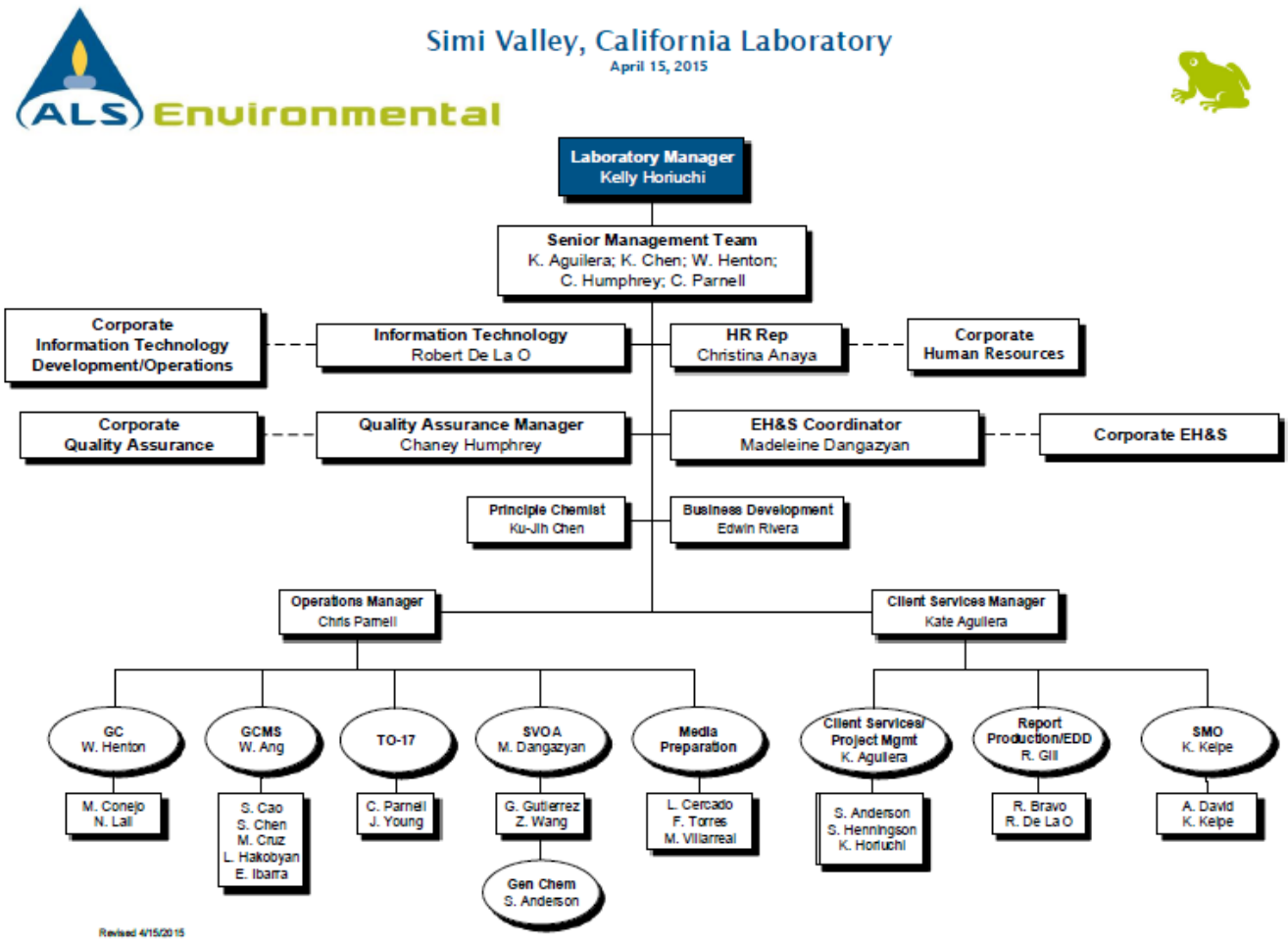
Acronym	Definition
AB	Accrediting Body
ACS	American Chemical Society
ANSI	American National Standards Institute
ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
BFB	4-Bromofluorobenzene
BTEX	Benzene, Toluene, Ethylbenzene, Xylenes
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service Registry Number
CCB	Continuing Calibration Blank sample
CCC	Continuing Calibration Check sample
CCV	Continuing Calibration Verification sample
CDC	Ongoing Demonstration of Capability
CLP	Contract Laboratory Program (through USEPA)
COC	Chain-of-Custody
DCM	Dichloromethane (aka Methylene Chloride)
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOC	Demonstration of Capability
DOE	Department of Ecology (state or federal)
DOH	Department of Health
EPA	U.S. Environmental Protection Agency (aka USEPA)
EPCRA	Emergency Planning & Community Right-to-Know Act
ERA	Environmental Resource Associates
ELAP	Environmental Laboratory Accreditation Program
FID	Flame Ionization Detector
FIFRA	Federal Insecticide, Fungicide & Rodenticide Act
FR	Federal Register
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HP	Hewlett-Packard (mfg. GC instruments)

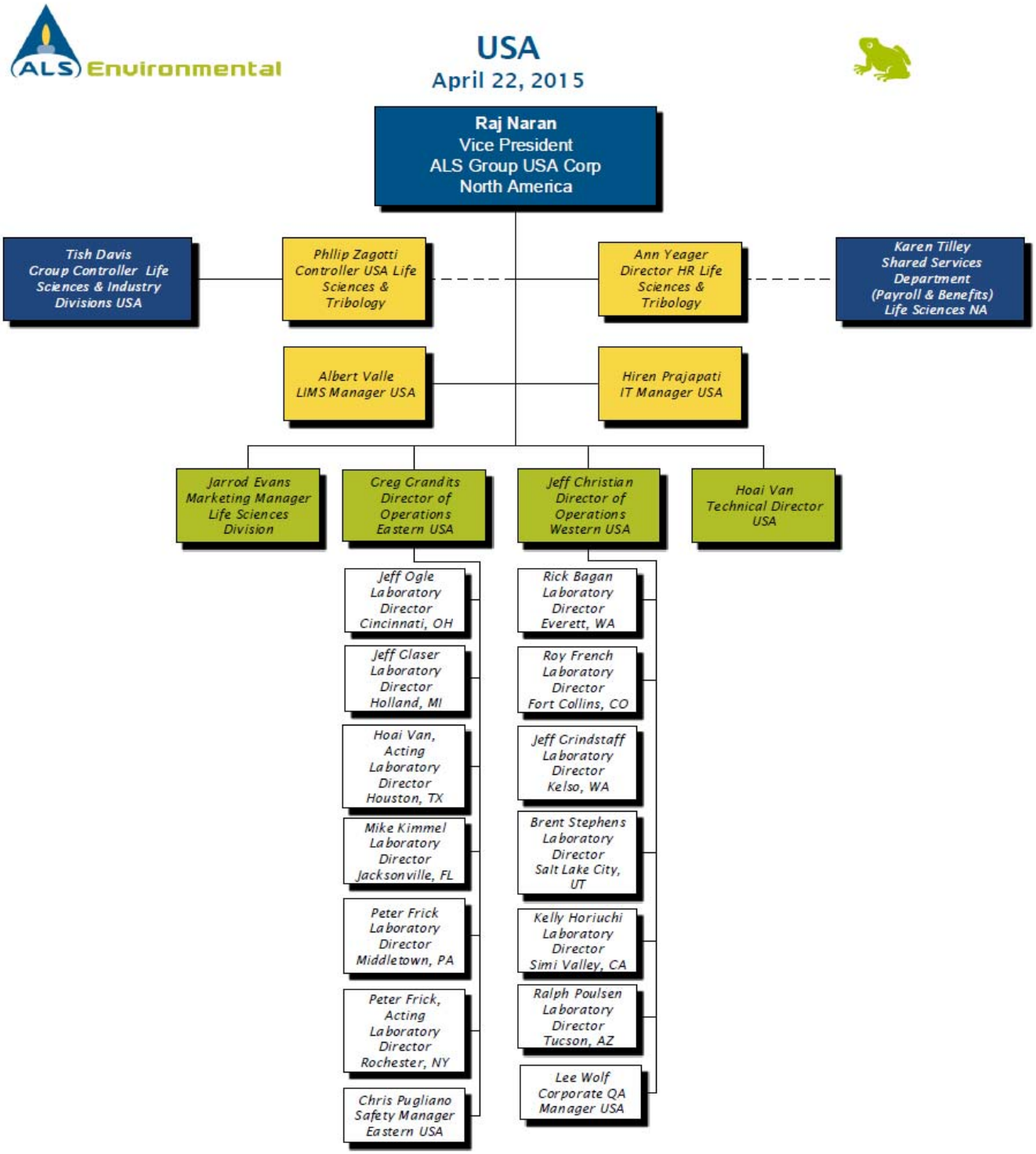
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICAL	Initial Calibration
ICB	Initial Calibration Blank sample
IDC	Initial Demonstration of Capability
ICV	Initial Calibration Verification sample
IFB	Invitation for Bid
ISO/IEC	International Organization for Standardization/International Electrochemical Commission
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LUFT	Leaking Underground Fuel Tank
MB	Method Blank
MDL	Method Detection Limit
MRL	Method Reporting Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NAS	National Academy of Sciences
NELAP	National Environmental Laboratory Accreditation Program
NCASI	National Council for Air and Stream Improvement (for the Paper Industry)
NCI	National Cancer Institute
ND	Not Detected
NIH	National Institute of Health
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NPD	Nitrogen Phosphorus Detector
NPDES	National Pollutant Discharge Elimination System
NSF	National Science Foundation
NTIS	National Technical Information System
NTP	National Toxicology Program
OSHA	Occupational Safety and Health Administration
PCBs	Polychlorinated Biphenyls
PE	Performance Evaluation sample
PID	Photoionization Detector

PQL	Practical Quantitation Limit
PT	Proficiency Test
QA	Quality Assurance
QAM	Quality Assurance Manual
QC	Quality Control
RAS	Routine Analytical Services (Contracts through USEPA)
RCRA	Resource Conservation and Recovery Act
RFP	Requests for Proposal
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SAS	Special Analytical Services (contracts through USEPA)
SIE	Selective Ion Electrode
SIM	Selected Ion Monitoring
SMO	Sample Management Office (aka Sample Receiving)
SOC	Semi-Volatile Organic Compounds
SOP	Standard Operating Procedure
SOQ	Statement of Qualifications
SOW	Statement of Work
SVOAs	Semi-Volatile Organic Analytes
SVOCs	Semi-Volatile Organic Compounds
SW-846	Test Methods for Evaluating Solid Waste, Physical/Chemical Methods
TNI	The NELAC Institute
TPH	Total Petroleum Hydrocarbons
TSCA	Toxic Substances Control Act
UST	Underground Storage Tank
UV	Ultraviolet Spectrophotometer
VOA	Volatile Organic Analyte
VOC	Volatile Organic Compounds
WP	Water Pollution
WS	Water Supply

Units	Definition
mg/kg	Milligrams per Kilogram
mg/L	Milligrams per Liter
mg/m3	Milligrams per Cubic Meter
ng/L	Nanograms per Liter
ppb	Parts Per Billion
ppbV	Parts Per Billion Volume
ppm	Parts Per Million
ppmV	Parts Per Million Volume
ug/L	Micrograms per Liter
ug/m3	Micrograms per Cubic Meter

APPENDIX B – Organization Charts and Key Personnel Qualifications





Kathleen 'Kate' Aguilera

2655 Park Center Drive, Suite A | Simi Valley, CA 93065 | +1 805 526 7161

ALS Environmental

Education

California State University - Northridge, CA
BA, Chemistry, 1989

Affiliations

American Chemical Society

Client Services Manager / Project Manager

2011 – Present

Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinates with the laboratory and administration to ensure that analyses are properly executed and meets the clients' needs.

Previous Experience

Columbia Analytical Services, Inc. — **Project Manager, '97 – '11**
Simi Valley, CA

Responsibilities: Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinates with the laboratory and administration to ensure that analyses are properly executed and meets the client's needs.

Columbia Analytical Services, Inc. — **GC/MS Analytical Chemist, '94 – '97**
(DBA Performance Analytical, Inc.)
Los Angeles, CA

Responsibilities: Analysis of air samples using EPA compendium methods TO-1, TO-2 and TO-14 using cryogenic concentration and thermal desorption techniques on whole air samples collected in summa canisters, Tedlar bags, and solid sorbent air samples. Proficient in the interpretation of mass spectra. Responsible for the preparation and quality control verification of solid sorbent sampling media for EPA Compendium methods TO-1 and TO-2.

Performance Analytical, Inc. — **GC/MS Analytical Chemist, '92 – '94**
Canoga Park, CA

Responsibilities: Analysis of air samples using EPA compendium methods TO-1, TO-2 and TO-14 using cryogenic concentration and thermal desorption techniques on whole air samples collected in summa canisters, Tedlar bags, and solid sorbent air samples. Proficient in the interpretation of mass spectra. Responsible for the preparation and quality control verification of solid sorbent sampling media for EPA Compendium methods TO-1 and TO-2.

Performance Analytical, Inc. — **GC Analytical Chemist, '89 – '92**
Canoga Park, CA

Responsibilities: Performed analyses of air samples for reduced sulfur compounds, hydrocarbon distribution and speciation, fixed atmospheric gases and total gaseous non-Methane organics. Performed analyses of soil and water samples for TPHg (mod. 8015) and BTEX. Performed extractions and analyses of CARB, NIOSH, OSHA and EPA 8000 series methods. Also performed metals analysis using flame and graphite furnace atomic absorption spectrophotometry (AA, GFAA).

Susan 'Sue' Anderson

2655 Park Center Drive, Suite A | Simi Valley, CA 93065 | +1 805 526 7161

ALS Environmental

Education

University of Illinois - Urbana-Champaign, IL
BS, Biochemistry, 1989

Project Manager/Technical Manager (General Chemistry)

2011 – Present

Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinates with the laboratory and administration to ensure that analyses are properly executed and meets the clients' needs. Also responsible for the training of general chemistry staff, maintenance of MDL studies, and standard operating procedures, data evaluation and report responsibility.

Previous Experience

Columbia Analytical Services, Inc. — **Project Manager/Technical Manager (General Chemistry), '06 – '11**
Simi Valley, CA

Responsibilities: Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinates with the laboratory and administration to ensure that analyses are properly executed and meets the client's needs. Also responsible for the training of general chemistry staff, maintenance of MDL studies, and standard operating procedures, data evaluation and report responsibility.

Columbia Analytical Services, Inc. — **Project Manager/Technical Manager (General Chemistry), '02 – '06**
Canoga Park, CA

Responsibilities: In addition to the Project Manager duties listed below, also responsible for the management of General Chemistry laboratory operations, including the financial aspects. This includes supervision and coordination of work load and training personnel as necessary as well as supervision of method development and certification, maintenance of MDL studies and SOPs, data evaluation and report responsibility. Other duties include participation in the formulation of project strategy and meetings involving major technical issues, working with regional senior management in short and long-range planning, and other duties as assigned.

Columbia Analytical Services, Inc. — **Project Manager II, '00 – '02**
Canoga Park, CA

Responsibilities: Responsibilities include interfacing with clients to provide technical project management and customer service, including project scheduling and tracking from the delivery of sample bottles to client site to the delivery of the completed analytical report. Ensures that the client receives timely, appropriate, and quality analytical services. Coordinates with the CAS laboratory and administration to ensure that analyses are properly executed and meet the clients' needs. Coordinates sub-contracting with internal and external laboratories. Acts as a liaison for all client-related activities within Columbia Analytical Services, Inc. Interfaces with work processing staff to answer technical questions that arise during EDD completion. Has high level role in data evaluation and report responsibility. High level client and regulatory agency contact.

Columbia Analytical Services, Inc. — **Scientist I-III, '92 – '00**
Canoga Park, CA

Responsibilities: Responsible for performing inorganic analyses such as: alkalinity, ammonia, BOD, COD, cyanide, sulfide, reactivity, fluoride, pH, hardness, hexavalent chromium, phenols, surfactants, total-dissolved-suspended solid, conductivity, turbidity, nitrate, chloride by titration, turbidimetric sulfate, color, odor, organic lead, residual chlorine, settleable solids, specific gravity, carbon dioxide, TCLP/STLC metals and semi-volatile extraction. Also perform analyses for TRPH and oil and grease and occasionally perform metals digestion. Also ran the Graphite furnace for all furnace metals and was responsible for standard prep and maintenance.

National Environmental Testing — **Wet Chemistry, '90 – '91**
Bartlett, IL

Responsibilities: Responsible for the analyses for wastewater parameters and some inorganic analytes.

Widayati 'Wida' Ang

2655 Park Center Drive, Suite A | Simi Valley, CA 93065 | +1 805 526 7161

ALS Environmental

Education

Technical University of West Berlin -
West Berlin, Germany
BS, Chemistry 1982

Technical University of West Berlin -
West Berlin, Germany
MS, Chemistry 1984

Volatile GC/MS Team Leader

2011 – Present

Team leader for the Volatile Gas Chromatography Mass Spectrometry Air group responsibilities are but are not limited to training of chemists, peer review of analytical data, mentoring of junior analysts, standard operating procedure review and streamlining of methods. Duties also require performance reviews and development of her direct reports.

Previous Experience

Columbia Analytical Services, Inc. — **Volatile GC/MS Team Leader, '08 – '11**
Simi Valley, CA
Duties as above.

Columbia Analytical Services, Inc. — **GC/MS Chemist, '07 – '08**
Simi Valley, CA
Analyzing indoor air, ambient air and source emission samples by GC/MS methods, standard preparation, perform maintenance on instruments when required, real time data reduction, participate in peer review process, and good practice of all QA/QC requirements.

Columbia Analytical Services, Inc. — **Technical Manager, Organic Chemistry, '99 – '07**
Canoga Park, CA
Responsible for managing the organics department with regards to State and Federal regulatory requirements. Supervises and coordinates work load and trained personnel. Supervised method development and certification, as well as method troubleshooting and instrument maintenance. Responsible for mobile laboratory operations.

Laboratory Data Consultants, Inc. — **Data Validator, '98 – '99**
Carlsbad, CA
Responsible for retrieving analytical data from closed down laboratory operations, review and validation of data packages. Supervised other employees for data package assembly

VOC Laboratories, Inc. — **Assistant QC Manager and Data Package Specialist, '96 – '98**
Glendale, CA
Responsible for overseeing data quality of final data validation packages. Managed production of data packages to meet various State and Federal analytical programs as well as customized client formats. Oversaw enforcement of the laboratory for implementation of corrective action measure. Interacted with chemists and project managers to ensure accuracy and completeness of data deliverables.

Thermo Analytical — **Technical Director/Department Manager, '92 – '96; Department Supervisor and Chemist, '88 - '92**
Monrovia, CA
Responsible for daily operations of the organic chemistry department. Developed standard operating procedures for various methods. Reviewed analytical data generated for completeness and contractual requirements according to Contract Laboratory Program (CLP) and SW-846 methods. Organized and scheduled reports for project managers. Responsible for upgrading and purchasing new instrumentation. Provided technical support to QC coordinator and laboratory personnel. Assisted with proposal preparation and audits. Responsible for training chemists and technicians in proper performance of various analytical methods. Responsible for sample analysis of water, soil, and air for volatile organics by GC and GC/MS. Assisted chemists in the analysis and interpretation of pesticides and PCBs.

Shankman Laboratories — **Analytical Chemist, '86-'88**
Los Angeles, CA
Prepared and analyzed soil and water samples using GC, GC/MS, HPLC, IR, IC and UV spectrophotometic techniques

Ku-Jih Chen

2655 Park Center Drive, Suite A | Simi Valley, CA 93065 | +1 805 526 7161

ALS Environmental

Education

National Chung-Hsing University - Taipei, Taiwan
BS, Botany, 1975

Principle Chemist

2011 – Present

Responsible for the development and validation of sampling and analysis methods, new technology and laboratory automation.

Previous Experience

Columbia Analytical Services, Inc.
Simi Valley, CA
Principle Chemist, '00 - '11
Responsibilities: Responsibilities listed above.

Columbia Analytical Services, Inc.
(dba Performance Analytical, Inc.)
Los Angeles, CA
Scientist VII, '94 - '00
Responsibilities: Responsibilities included operating the gas chromatography and sample preparation laboratories, developing methods (previously developed the Total Combustion Analyzer for the measurement of reactive organic gases in stationary source samples, and the Determination of Reduced Sulfur Compounds and fixed atmospheric gases in POTW emissions, refinery and landfill gases), and serving as the laboratory's primary Industrial Hygiene Chemist.

Performance Analytical, Inc.
Canoga Park, CA
Principle Chemist, '89-'94
Responsibilities: Responsibilities listed above.

C-E Environmental, Inc.
Camarillo, CA
Extraction Laboratory Supervisor, '84 – '89
Responsibilities: Responsibilities included supervising chemists, associate chemists, and technicians, preparing SOPs, analytical standards, and spiking solutions, serving as Primary Extraction Chemist for the Love Canal Habitability Study, and previously responsible for instrumental analysis using GC, LC, GC/MS, and AA.

Paolyta Company
Taipei, Taiwan
Research and Development Chemist, '80 – '84

Panlabs Taiwan Ltd.
Taipei, Taiwan
Research Chemist, '75 – '80

Madeleine Dangazyan

2655 Park Center Drive, Suite A | Simi Valley, CA 93065 | +1 805 526 7161

ALS Environmental

Education

California State University at Northridge - Northridge, CA
BS, Chemistry 1995

Semi-Volatiles Technical Manager and EH&S Manager

2011 – Present

As EH&S Manager, is responsible for the implementation of the Environmental Health and Safety program of ALS North America to this facility. Duties include accident investigation and incident review, maintenance of all safety-related equipment and documents, and performing safety audits and reporting results to management. Semi-Volatiles/Industrial Hygiene Technical Manager responsibilities are but not limited to training of junior chemists, data reduction and peer review of analytical data, mentoring of junior analysts, writing and reviewing of standard operating procedures. Development and implementation of new methods. Duties also require performance reviews and development of direct reports. Additional responsibilities are analyzing ambient air, source emissions, and industrial hygiene samples using GC and HPLC utilizing OSHA, NIOSH and EPA mandated methodologies. Preparation and analysis of air samples taken on various sorbent tubes for semi-volatile organic compounds. Determination of Carbonyls, Phenols and Cresols in ambient air and source emission samples using HPLC. Determination of Polynuclear Aromatic Hydrocarbons using EPA Method TO-13A. Analysis of Pesticides and PCBs using EPA Methods TO-4A and TO-10A. Routine and necessary instrument maintenance.

Previous Experience

Columbia Analytical Services, Inc. — **Semi-Volatiles Technical Manager, '02- '11; EH&S Manager '10-11**
Simi Valley, CA

Responsibilities: Responsibilities listed above.

Columbia Analytical Services, Inc. — **Scientist, '00- '02**
Simi Valley, CA

Responsibilities: Responsibilities include training of chemists, peer review of analytical data, mentoring or junior analyst, standard operating procedure review, and streamlining of methods. Additional responsibilities are analyzing ambient air, source emissions, and industrial hygiene samples using GC and HPLC utilizing OSHA, NIOSH and EPA mandated methodologies. Preparation and analysis of air samples taken on various sorbent tubes for semi-volatile organic compounds. Determination of Carbonyls, Phenols and Cresols in Aromatic Hydrocarbons using EPA Method TO-13A. Analysis of Pesticides and PCBs using EPA Methods TO-4A and TO-10A. Routine and necessary instrument maintenance.

Columbia Analytical Services, Inc. — **Scientist, '99- '00**
(dba Performance Analytical, Inc.)
Simi Valley, CA

Responsibilities: Responsibilities include analyzing indoor and ambient air, source emission, and industrial hygiene samples by GC methods.

Air Products and Chemicals, Inc. — **Analytical Chemist, '95 – '99**
Long Beach, CA

Responsibilities: Quality assurance analysis of EPA protocol gases utilizing GC, FTIR and NDIR. Preparation of personnel schedules, lead laboratory contact.

California State University at Northridge — **Undergraduate Researcher, '93 – 94**
Northridge, CA

Responsibilities: Assisted professor with improving and implementing student laboratory experiments to better utilize a GC/MS.

Wade Henton

2655 Park Center Drive, Suite A | Simi Valley, CA 93065 | +1 805 526 7161

ALS Environmental

Education

University of California at Santa Barbara – Santa Barbara, CA
BS, Chemistry 1985

Volatile GC Team Leader

2011 – Present

Team leader for the Volatile Gas Chromatography department where responsibilities include but are not limited to training of chemists, peer review of analytical data, mentoring of junior analysts, standard operating procedure review, and streamlining of methods. Duties also require performance reviews and development of direct reports.

Previous Experience

Columbia Analytical Services, Inc. — **Volatile GC Team Leader, '00 – '11**
Simi Valley, CA
Responsibilities listed above.

Columbia Analytical Services, Inc. — **Scientist V, '95 – '00**
(dba Performance Analytical, Inc.)
Los Angeles, CA
Responsibilities include analyzing indoor and ambient air, source emission, and industrial hygiene samples by GC and GC/MS methods.

Columbia Analytical Services, Inc. — **Scientist IV, '94 – '95**
(dba Performance Analytical, Inc.)
Los Angeles, CA
Responsibilities listed above.

Coast-to-Coast Analytical Services — **Analytical Chemist, '92 – '94**
Camarillo, CA
Responsibilities included analyzing samples using EPA methods 625, 525 and 1625 as well as developing new methods for GC/MS testing.

Coast-to-Coast Analytical Services — **Analytical Chemist, '91 – '92**
Goleta, CA
Responsibilities included analyzing samples using EPA methods 624 and 524.2 by GC/MS. Used GC/MS methods to perform fuel fingerprinting

Combustion Engineering Environmental — **Analytical Chemist, '86 – '91**
Camarillo, CA
Responsibilities included method development for GC and HPLC. Analysis of samples using EPA methods 608, 615, 631, 632 and SW846. Other methods used include 8080, 8010, 8020, 8150 and 8030. Oversaw data integrity for the GC Laboratory instrument data network. Data review.

Fortin Industries — **Chemist, '86**
Sylmar, CA
Research and Development and Quality Assurance/Quality Control on polymer products and metal coatings using differential scanning calorimeters, scanning electron microscope, AA, GC, and HPLC.

Kelly M. Horiuchi

2655 Park Center Drive, Suite A • Simi Valley, CA 93065 • +1 805 526 7161

ALS Environmental

Education

California State University, Northridge,CA
BA, Biology, 1998

Laboratory Director

2011 - Present

Primary responsibilities include management of all laboratory departments, scheduling, productivity, reporting and evaluation of analytical methodologies, project planning, budgeting, and Quality Assurance/Quality Control protocol oversight. Other responsibilities include conducting facility compliance reviews; providing departmental support for equipment purchases; resolving personnel issues; determining resource allocation; and providing supervision, training, and leadership to key laboratory staff. In addition, other responsibilities include direct responsibility for national contracts and consultants.

Previous Experience

Columbia Analytical Services, Inc.
Simi Valley, CA
Laboratory Director, '09-'11

Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance.

Columbia Analytical Services, Inc.
Simi Valley, CA
Project Manager, '05-'09

Responsibilities: Interfacing with clients to provide technical project management and customer service, including project scheduling, tracking and consulting to determine appropriate sampling and analytical protocols. Coordinated with the laboratory and administration to ensure that analyses were properly executed and meets the client's needs.

Columbia Analytical Services, Inc.
Simi Valley, CA
Data Validation Coordinator,'03-'05

Responsibilities: Validation of analytical results produced by the laboratory. Verification of client analytical requests, sample information, and reporting formats. Interacts with project managers and Quality Assurance Program Manager to ensure that all reports fulfill client requirements as well as QA/QC needs. Compiled quality control summary, and calibration data upon client request for data packages. Assist the Quality Assurance Program Manager with standard operating procedures, control charting, and audit preparation.

Cure Autism Now
Los Angeles, CA
Database Analyst, '02-'03

Responsibilities: Performed analysis of test date through data audits and queries, maintained extensive database, and coordinated data audits between Northern and Southern California locations. Additional duties included assisting in the creation of new databases, as needed, creation of SOP for phenotypic and genotypic data collecting, and process improvements for subject flow through the research project.

Columbia Analytical Services, Inc.
(dba Performance Analytical, Inc.)
Simi Valley, CA
Scientist II, Data Validation Coordinator, '00-'02

Responsibilities: Validation of all analytical results produced by the laboratory. Verification of client analyses, sample information, and reporting format. Compiled quality control summary, and calibration data upon client request for data packages. Assisted the Quality Assurance Program Manager with standard operating procedures, control charting, and audit preparation.

Specialty Laboratories
Santa Monica, CA
Administrative Assistant, Data Analyst, '99-'00

Responsibilities: Performed retrieval, quality control, and organization of data. Compiled data for reporting of HIV, lead, urinalysis, kidney stones, and communicable diseases. Also communicated with the state DOH and clients regarding reporting requirements and demographic information.

Chaney Humphrey

2655 Park Center Drive, Suite A • Simi Valley, CA 93065 • +1 805 526 7161

ALS Environmental

Education

Oregon State University,
Corvallis, OR
BS, Biology, 2004

Quality Assurance Manager

2011 - Present

Responsibilities include facilitate ethics and QA training, maintain all training documentation, perform QA orientation for new employees, review data (both hardcopy and electronic), perform internal QA audits and prepare written reports, review, approve, and control Standard Operating Procedures, maintain QA Manual, maintain QA records (including archived logbooks, archived certificates of analysis, nonconformity and corrective action reports, MDL studies results, SOP revision and distribution, statistical control limits, PE sample results), serve as document control officer, and PC for all PE sample analyses, prepare corrective action report for any unacceptable PE sample results, maintain laboratory's certifications and approvals, facilitator for external QA audits and prepare written response to deficiencies, prepare activity report to management.

Previous Experience

Columbia Analytical Services, Inc. — **Quality Assurance Manager, -09 -11**
Simi Valley, CA

Duties same as above.

Columbia Analytical Services, Inc. — **Data Validation Coordinator, '07-'09**
Simi Valley, CA

Responsibilities include validation of analytical results produced by the laboratory. Verification of client analytical requests, sample information, and reporting formats. Interacts with project managers and Quality Assurance Program Manager to ensure that all reports fulfill client requirements as well as QA/QC needs. Compiled quality control summary, and calibration data upon client request for data packages.

Columbia Analytical Services, Inc. — **GC/MS Chemist,'05-'07**
Simi Valley, CA

Responsibilities: Analyzing indoor air, ambient air and source emission samples by GC/MS methods, standard preparation, perform maintenance on instruments when required, real time data reduction, participate in peer review process, and good practice of all QA/QC requirements.

Columbia Analytical Services, Inc. — **Analyst, '04-'05**
Kelso, WA

Responsibilities: Performed a variety of analytical tests within the General Chemistry laboratory according to EPA Methodologies including Ion Chromatography, total sulfur, and solids. Saturday crew member responsible for performance of all short hold time methods including microbiology methodologies.

Columbia Analytical Services, Inc. — **Temporary Employee, Summers '02-'04**
Kelso, WA

Responsibilities: Temporary employee (summers) performing a variety of analytical tests including grain size, total organic carbon, total suspended solids, total dissolved solids, alkalinity, acidity, and chemical oxygen demand. Additionally, performed colorimetric methods including ortho-phosphorous, total-phosphorous, hexavalent chromium, and nitrite as nitrogen.

Christopher Parnell

2655 Park Center Drive, Suite A | Simi Valley, CA 93065 | +1 805 526 7161

ALS Environmental

Education

University of California
at Santa Barbara
Santa Barbara, CA
BS, Chemistry 1986

Operations Manager/Technical Advisor (Volatile GC/MS Air)

2012 – Present

Operation Managers responsibilities include planning, directing, and coordinating the operations of the laboratory departments. Duties and responsibilities include formulating policies, managing daily operations, and planning the use of materials and human resources. Reviews performance data to measure productivity and goal achievement and to determine areas needing cost reduction and program improvement to increase efficiency.

Technical Advisor for the Volatile Gas Chromatography Mass Spectrometry department. Has the responsibility of oversight of training of chemists, peer review of analytical data, mentoring of junior analysts, standard operating procedure review and streamlining of methods. Duties also require performance reviews and development of direct reports.

Previous Experience

ALS Environmental
Simi Valley, CA
Technical Advisor (Volatile GC/MS Air), '11 – '12
Responsibilities: Technical Advisor responsibilities listed above.

Columbia Analytical Services, Inc.
Simi Valley, CA
Technical Advisor (Volatile GC/MS Air, '08 – '11
Responsibilities: Technical Advisor responsibilities listed above.

Columbia Analytical Services, Inc.
Simi Valley, CA
GC/MS Team Leader, '00 – '08
Responsibilities: Team leader for the Volatile Gas Chromatography Mass Spectrometry group. Responsibilities include training of chemists, peer review of analytical data, mentoring of junior analysts, standard operating procedure review, and streamlining of methods. Duties also require performance reviews and development of direct reports.

Columbia Analytical Services, Inc.
(dba Performance Analytical, Inc.)
Los Angeles, CA
Scientist VI, '94 – '00
Responsibilities: Responsibilities include analyzing indoor air, ambient air and source emission samples by GC/MS methods, standards preparation, perform maintenance on instruments when required, real time data reduction, participation in peer review process, and good practice of all QA/QC requirements.

Performance Analytical, Inc.
Canoga Park, CA
Scientist VI, '91 – '94
Responsibilities: Responsibilities listed above.

ABB Environmental Inc.
Camarillo, CA
Air Toxics Laboratory Supervisor, '90 – '91
Responsibilities: Responsibilities included scheduling client analyses and developing methods for non-routine analyses, and operating the Air Toxics laboratory.

C-E Environmental Inc., EMSI
Camarillo, CA
Analytical Chemist, '87 – '90
Responsibilities: Responsibilities included overseeing the Pesticide/PCB analysis of samples under the EPA Contract Laboratory Program, and interfacing with the EPA and regional offices to respond to inquiries, and performing GC analyses and extractions.

Damon Reference Laboratory
Newbury Park, CA
Chemist, '86 – '87
Responsibilities: Responsibilities included performing Enzyme-linked immunosorbent assays, Western-Blot assays, and Protein Electrophoresis.

APPENDIX C – Ethics and Data Integrity Policy

ETHICS AND DATA INTEGRITY AGREEMENT

I state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at ALS.

I agree that in the performance of my duties at ALS:

1. I shall not intentionally report data values that are not the actual values obtained;
2. I shall not intentionally report the dates, times and method citations of data analyses that are not the actual dates, times and method citations of analyses;
3. I shall not intentionally represent another individual's work as my own;
4. I shall not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operating Procedures, or as defined by company policy.
5. I agree to inform ALS of any accidental or intentional reporting of non-authentic data by other employees.
6. I have read this ethics and data integrity agreement and understand that failure to comply with the conditions stated above will result in disciplinary action, up to and including termination.
7. I agree to adhere to the following protocols and principals of ethical conduct in my work at ALS. All work assigned to me will be performed using ALS approved methods and procedures and in compliance with the quality assurance protocols defined in the ALS Quality System.
8. I will not intentionally falsify nor improperly manipulate any sample or QC data in any manner. Furthermore, I will not modify data values unless the modification can be technically justified through a measurable analytical process or method acceptable to ALS. All such modifications and their justification will be clearly and thoroughly documented in the raw data and appropriate laboratory record, and will include my initials or signature and the date.
9. I will not make false statements to, or seek to otherwise deceive ALS staff, managers or clients. I will not knowingly, through acts of commission, omission, erasure or destruction, improperly report any test results or conclusions, be they for client samples, QC samples, or standards.
10. I will not condone any accidental or intentional reporting of unauthentic data by other ALS staff and will immediately report such occurrences to my Supervisor, Lab Director, Quality Assurance Manager, or Human Resources. I understand that failure to report such occurrences may subject me to immediate discipline, including termination.
11. If a supervisor, manager, director or other member of the ALS leadership group requests me to engage in or perform an activity that I feel is compromising data validity or defensibility, I have the right to not comply with the request. I also have the right to appeal this action through an ALS local Quality Staff, Corporate Quality Assurance or Human Resources.
12. I understand that if my job includes supervisory responsibilities, I will not instruct, request or direct any subordinate to perform any unethical or non-defensible laboratory practice. Nor will I discourage, intimidate or inhibit a staff member who may choose to appropriately appeal my supervisory instruction, request or directive that may be perceived to be improper, nor retaliate against those who do so.
13. I understand that employees who report violations of this policy will be kept free from intimidation and recrimination arising from such reporting.

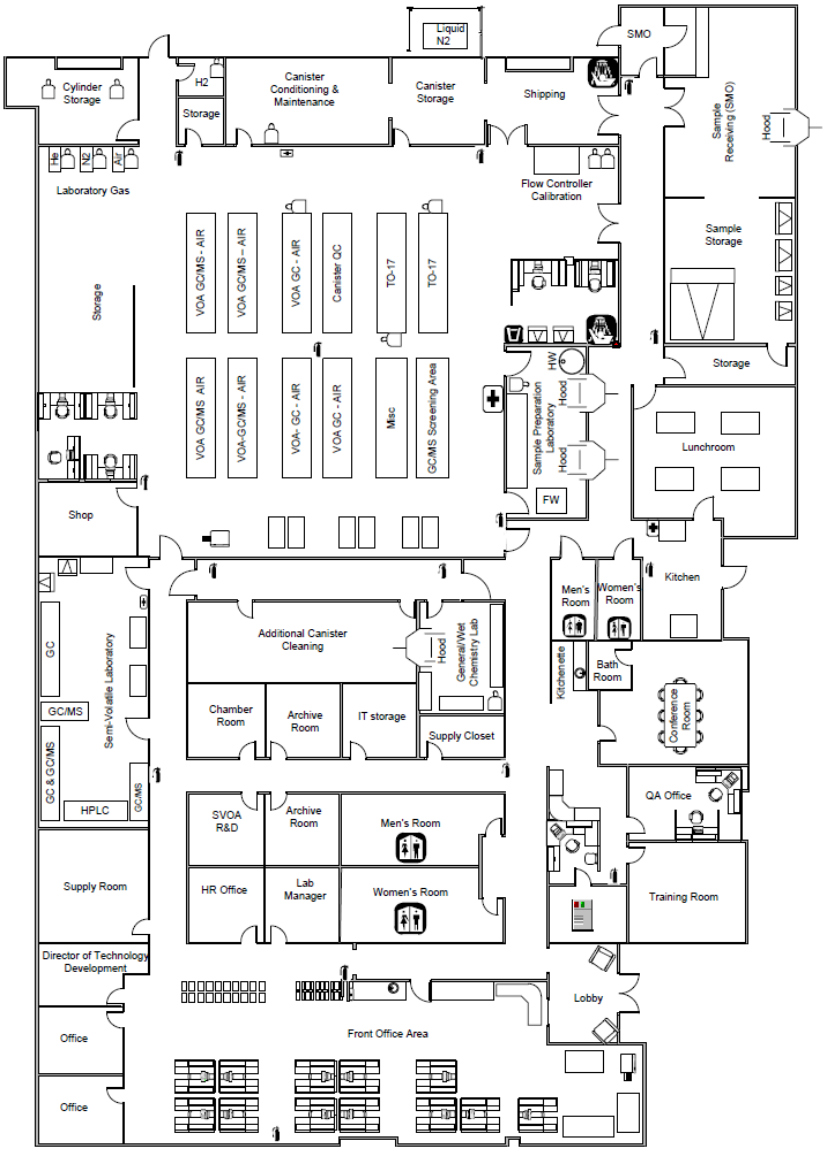
I have read, and understand the above policy and realize that failure to adhere to it may result in disciplinary action, up to and including termination. Compliance with this policy will be strictly enforced with all personnel employed by the company.

Employee Name _____ Signature _____

ALS Location_____ Date _____

APPENDIX D – Laboratory Floor Plan

ALS Environmental-Simi Valley Laboratory Floor Plan

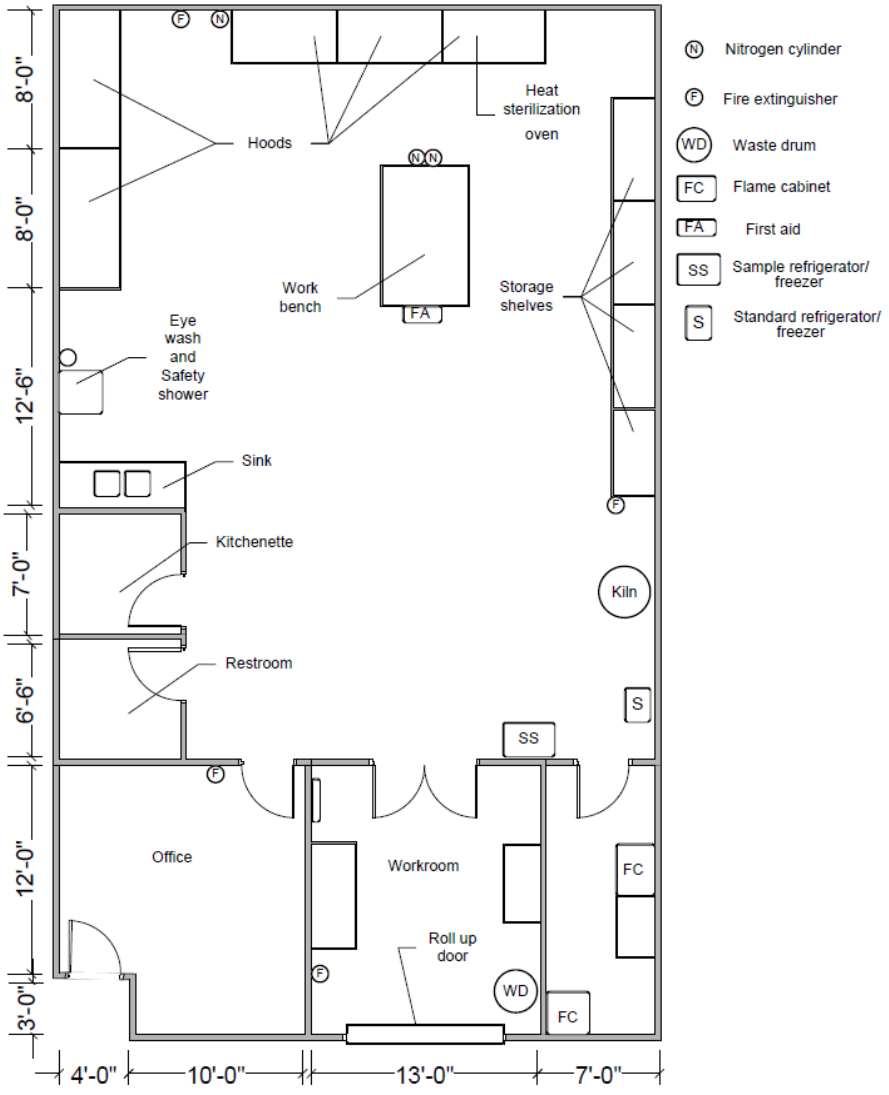


ALS ENVIRONMENTAL - SIMI VALLEY FLOOR PLAN		
2655 Park Center Drive, Suite A, Simi Valley, California 93065		
-First Aid	HW -Hazardous Waste Cabinet	
-Network Server Room	-Emergency Shower	FW -Flammable Waste Cabinet
-Fire Extinguisher	-Gas Cylinder(s)	
-Refrigerator/Freezer	-Deionized Water	

ALS Environmental-Simi Valley Extraction Laboratory Floor Plan

Extraction Laboratory for
ALS Environmental

2360 Shasta Way, Unit G
Simi Valley, CA. 93065



APPENDIX E – Analytical Equipment

Equipment Description - Gas Chromatography	Purchased / Acquired	Location
Screen 02: Hewlett-Packard 5890 with FID Detector	-	VOA GC/MS Screen
Screen 03: Hewlett-Packard 5890 with FID Detector	-	VOA GC/MS Screen
GC01: Hewlett-Packard 5890 with FID/TCD Detectors *Fixed Gas Analyzer/Total Combustion Analyzer (TCA)*	1995	VOA GC
GC03: Hewlett-Packard 5890 with ECD/FID Detectors *Hewlett-Packard 7673 Autosampler*	1995	SVOA
GC05: Hewlett-Packard 5890 Series II Combined with Sievers 355 (SCD 1)	1996	SVOA
GC06: Hewlett-Packard 6890 with ECD/ECD Detectors *Hewlett-Packard 6890 Autosampler*	1995	SVOA
GC07: Hewlett-Packard 6890 with FID/FID Detectors	1995	VOA GC
GC08: Hewlett-Packard 5890 Series II with TCD/FID Detectors	1998	VOA GC
GC09: Hewlett-Packard 5890 Series II with FID Detector	1999	VOA GC/MS Screen
GC10: Hewlett-Packard 5890A with FID/TCD Detectors	1999	VOA GC
GC11: Hewlett-Packard 5890 Series II+ with FID Detector (Combined with MS01)	1999	SVOA
GC12: Hewlett-Packard 5890 Series II+ with FID Detector (Combined with MS02)	2004	SVOA
GC13: Agilent 6890A Combined with Sievers 355 (SCD 2)	2001	VOA GC
GC14: Agilent 6890N with NPD/FID Detectors *Agilent 7683B Autosampler*	2005	SVOA
GC15: Agilent 6890N with NPD/FID Detectors *Agilent 7683 Autosampler*	2005	SVOA
GC16: Agilent 6890N with PFPD Detector and *OI Detector Controller* *Agilent 7683 Autosampler*	2005	SVOA
GC19: Hewlett-Packard 5890 with FID Detector	2007	VOA GC
GC20: Agilent 7890A with FID/TCD Detectors	2008	VOA GC
GC21:Hewlett-Packard 5890 Series II with ECD/FID Detectors	2009	SVOA
GC22: Agilent 7890A Combined with Agilent 355 (SCD 3)	2009	VOA GC
GC23: Hewlett-Packard 6890+ with ECD Detector (Combined with MS14)	2007	SVOA
GC24: Hewlett-Packard 5890 Series II (Combined with MS04)	2011	VOA GC
GC25: Hewlett-Packard 5890 Series II (Combined with MS12)	2006	SVOA
GC26: Agilent 7890A (Combined with MS19)	2011	VOA GC/MS
GC27: Agilent 7890A (Combined with MS20)	2011	VOA GC/MS

Equipment Description - GC/MS Systems	Purchased / Acquired	Location
MS01: HP 5890 Series II+ with FID Detector (GC11) & HP 5971A MSD *Hewlett-Packard 7673 Autosampler*	1991	SVOA
MS02: HP 5890 Series II+ with FID Detector (GC12) & HP 5972 MSD *Hewlett-Packard 7673 Autosampler*	1994	SVOA
MS04: HP 5890 Series II (GC24) & HP 5970 MSD	2004	VOA GC
MS05: Agilent 6890+/5973N MSD *Perkin Elmer TurboMatrix ATD-50 Thermal Desorber*	1999	VOA GC/MS
MS07: HP 6890A/ Agilent 5973N MSD	2001	SVOA
MS08: Agilent 6890N/5973inert MSD Tekmar AUTOCAN Autosampler	2004	VOA GC/MS
MS09: Agilent 6890N/5973inert MSD Tekmar AUTOCAN Autosampler	2005	VOA GC/MS
MS10: HP 6890A/5973 MSD	2006	SVOA
MS11: HP 5890 Series II/5972 MSD	2006	SVOA
MS12: HP 5890 Series II (GC25)/5971 MSD HP 7673 Autosampler	2006	SVOA
MS13: Agilent 6890N/5975B inert MSD Tekmar AUTOCAN Autosampler	2006	VOA GC/MS
MS14: HP 6890+ with ECD Detector (GC23) & HP 5973 MSD HP 6890 Injector	2007	SVOA
MS15: HP 5890 Series II/5972 MSD HP 7673 Autosampler	2007	SVOA
MS16: Agilent 6890N/5975C inert MSD Tekmar AUTOCAN Autosampler	2007	VOA GC/MS
MS17: Shimadzu GCMS QP-2010 Plus	2008	VOA GC/MS
MS18: Agilent 7890A /5975C inert XL MSD Markes Series 2 Unity Thermal Desorber Markes Series 2 Ultra TD Autosampler	2010	VOA GC/MS
MS19: Agilent 7890A (GC26) & 5975C inert XL MSD Tekmar AUTOCAN Autosampler	2011	VOA GC/MS
MS20: Agilent 7890A (GC27) & /5975C inert XL MSD Markes Series 2 Unity Thermal Desorber Markes Series 2 Ultra TD Autosampler	2011	VOA GC/MS
MS21: Agilent 7890A (GC28) & 5975C inert XL MSD Tekmar AUTOCAN Autosampler	2012	VOA GC/MS
MS22: Agilent 7890B (GC29) & 5977A MSD Markes CIA Advantage Autosampler	2015	VOA GC/MS

Liquid Chromatography	Purchased / Acquired	Location
LC03: Agilent Infinity LC 1220 (Combined with LCMS01)	2011	SVOA
LCMS01: Agilent 6120 Quadrupole MS (Combined with LC03)	2011	SVOA
Ion Chromatography	**Purchased / Acquired**	**Location**
IC03: Dionex ICS 2000 with Self-regenerating suppressor AS40 Autosampler	2008	GENCHEM
Spectrophotometer	**Purchased / Acquired**	**Location**
SPM01: Spectronic Instrument 20+ from SC	2001	GENCHEM
pH and Specific Ion Meters	**Purchased / Acquired**	**Location**
pH01: Thermo Orion 920 Selective Ion Meter	2001	GENCHEM
pH02: Orion 720A	1992	GENCHEM
Miscellaneous Equipment	**Purchased / Acquired**	**Location**
US Filter Water Purification System	2006	Main Lab
US Filter Water Purification System	2008	Extraction facility

Note: Purchase / Acquired year may represent when instrument was first maintained by ALS Environmental-Simi Valley or other in-network ALS Laboratory and does not reflect age of instrument.

Air sampling containers / Flow Controllers / Critical Orifices

Six-liter Summa passivated stainless steel canisters

- 1049 Ambient
- 1234 Source
- 191 Standard

Six-liter Silonite passivated stainless steel canisters

- 791 Ambient
- 334 Source

Three-liter Silco passivated stainless steel canisters (71)

One-liter Summa passivated stainless steel canisters (1112)

One-liter Silonite passivated stainless steel canisters (189)

400-millilter mini passivated stainless steel canisters (18)

Low volume flow controllers for time integrated sampling

- 863 Ambient
- 125 Source

Low-flow flow controllers for multi-day sampling (61)

Mini-canister flow controllers for time integrated sampling (16)

Critical orifices (2102)

Critical orifices – Sulfur (171)

Automated Summa Canister Conditioning Units

- Twenty-four position, microprocessor controlled conditioners with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (2)
- Ten position, microprocessor controlled conditioners with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (2)
- Fourteen position, microprocessor controlled conditioners with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (1)
- Sixteen position, microprocessor controlled conditioner with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (2)
- Six position, microprocessor controlled conditioner with heater controller, vacuum gauge, humidified nitrogen fill capability and large-capacity vacuum pump (1)

APPENDIX F – Containers, Preservation and Holding Times

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Maximum Holding Time
Solid / Water Sample Analysis				
Bromide (EPA 9056)	S,W	P, FP, G	Cool, 4˚C	28 Days
Chloride (EPA 9056)	S,W	P, FP, G	None Required	28 Days
Fluoride (9056)	S,W	P	Cool, 4˚C	28 days
Hydrogen Ion - pH (EPA 9040B/9045C)	S,W	P, FP, G	None Required	Analyze immediately
Nitrate, Nitrite (EPA 9056)	S,W	P, FP, G	Cool, 4˚C	48 hours
Orthophosphate (EPA 9056)	S	P,G	Cool, 4˚C	48 hours
Formaldehyde, Acetaldehyde (EPA 8315A Procedure 1 Modified)	S,W	Glass w/Teflon-Lined Lid	Cool, 4˚C	Aqueous – prep. - 72 hours, analysis – 30 days; Soil – prep. minimum, analysis – 30 days
Copper Corrosion (In-House Method)	Solid Wallboard	Ziploc Bag, G	None Required	-
H2S/Sulfur Emission (In-House Method)	Solid Wallboard	Ziploc Bag, G	None Required	-
Orthorhombic Cyclooctasulfur (In-House Method)	Solid Wallboard	Ziploc Bag, G	None Required	-

* W = Water or Aqueous solution; S = Soil or Sediment; P = Polyethylene, G = Glass, FP = fluoropolymer

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Maximum Holding Time	Sample Vol.[c]
Air Corrosivity	Air	Air Corrosivity Probes	Include 3 small dessicant bags (or equivalent) to each probe vial during shipment.	N/A[d]	3 Day Minimum Exposure
Amines (In-House Method)	Air	Treated Alumina Tubes	Sample Receipt-NA; Storage 4˚C±2˚C	30 days	100L
Ammonia (OSHA ID-188/ID-164)	Air	H_2SO_4 Treated Carbon Bead Tubes	Sample Receipt-NA; Storage 4˚C±2˚C	28 days	TWA: 24L STEL: 7.5L
BTU by ASTM D 3588 (SULFUR, ASTM D 5504; C1-C6+, EPA TO-3M; FIXED GASES, 3C)	Gaseous Fuels	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	<u>Sulfur</u> Bag – 24 hours Canister – 7 days[b] Bottle Vac[a] – 7 days[b] <u>C1-C6+</u> Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b] <u>3C</u> Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Carboxylic Acids (In-House Method)	Air	Treated Silica Gel Tubes	Sample Receipt-NA Storage 4˚C±2˚C	30 days until extraction; 14 days for analysis	100L
Total Gaseous Non-methane Organics (TGNMO) (EPA 25C)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Fixed Gases (EPA 3C & ASTM D 1946)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Helium & Hydrogen (EPA 3C Modified)	Air	Summa Canister Bottle Vac	N/A	Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Maximum Holding Time	Sample Vol.[c]
Argon (EPA 3C Modified)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours[b] Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Methane, Ethane, Ethene, Propane, Propene (RSK 175)	Aqueous	Glass w/Teflon- Lined Lid	No Headspace HCl to pH<2 4˚C±2˚C	14 days when preserved	(3) 40mL Vials
Carbon Dioxide (RSK 175)	Aqueous	Glass w/Teflon Lined Lid	No Headspace neutral pH (5-8) 4˚C±2˚C	N/A[d]	(3) 40mL Vials
Sulfur Compounds (In-House Method)	Aqueous	Glass w/Teflon Lined Lid	No Headspace; pH>4; 4˚C±2˚C	Following pH adjustment – 24 hours	(2) 40mL Vials
Sulfur Compounds (ASTM D 5504; SCAQMD 307-91; Modified SCAQMD 307-91)	Air	Tedlar Bag Fused Silica Lined Stainless Steel Canister Bottle Vac	No direct sunlight	Bag – 24 hours Canister – 7 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
C_1-C_6+ (EPA TO-3 Modified)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Methanol, Ethanol, Isopropyl alcohol, Freon, and Methylene Chloride (EPA TO-3 Modified)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Total Petroleum Hydrocarbons (TPHG) (EPA TO-3 Modified)	Air	Tedlar Bag Mylar Bag Summa Canister Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters and Bottle Vacs ≥1.0L
Pesticides and Polychlorinated Biphenyls (PCBs) (EPA TO-4A & TO-10A)	Air	Glass PUF Cartridge; TO-4A (High Volume); TO-10A (Low Volume)	Sample Receipt, <4˚C; Store sample and extract at <4˚C	7 days until extraction; extract – 40 days	2 m^3

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Maximum Holding Time	Sample Vol.[c]
Formaldehyde & Other Carbonyl Compounds (EPA TO-11A)	Air	DNPH-Coated Silica Gel Cartridge w/ Polypropylene Cap; SKC UME[x] and Bacharach GMD 570 Passive Monitors (formaldehyde only)	Sample Receipt, 4˚C±2˚C; Laboratory Preservation, 4˚C±2	14 days until extraction; 30 days for analysis	100 – 150L
Polycyclic Aromatic Hydrocarbons (PAHs) (EPA TO-13A)	Air	Polyurethane Foam (PUF) plugs, XAD Tube, PUF / XAD-2	Sample Receipt, <4˚C; Laboratory Preservation, <4˚C	7 days until extraction; 40 days after	130 – 400 m³
Volatile Organic Compounds (EPA TO-14A & TO-15)	Air	Tedlar Bag, Summa Canister (1L, 6L) Bottle Vac	N/A	Bag – 72 hours Canister – 30 days Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters 1.0L/6.0 Bottle Vacs 1.0L
Volatile Organic Compounds (EPA TO-17)	Air	Sorbent Tubes w/Swagelock Caps & PTFE Ferrules	<4˚C; organic solvent free environment; Laboratory Storage, 4˚C±2˚C	30 days	1-4L
Air-Phase Petroleum Hydrocarbons (MADEP APH)	Air	Summa Canister Bottle Vac	N/A	28 days Bottle Vac[a] – 30days[b]	Canisters 1.0L/6.0 Bottle Vacs 1.0L
Halogenated Volatile Organic Compounds (CARB 422)	Air	Tedlar Ba Summa Canister (1L, 6L) Bottle Vac	N/A	Bag – 72 hours Canister[a] – 30 days[b] Bottle Vac[a] – 30 days[b]	Bags 500mL Canisters 1.0L/6.0L Bottle Vacs 1.0L
Organic Vapors / NAPHTHAS (Diesel; etc.) (NIOSH 1550 / OSHA 7)	Air	Charcoal Tube; 3M 3500 or 3520 Badge; Silica Gel Tube w/ plastic caps	Sample Receipt-NA; Storage 4˚C±2˚C	14 days	Various
Sulfur Hexafluoride (NIOSH 6602 Modified)	Air	Tedlar Bag Summa Canister (1L, 6L)	N/A	Bag[b] – 72 hours Canister[a] – 30 days[b]	Bags 500mL Canisters 1.0L/6.0L
Siloxanes (In-House Method)	Air	SPE Cartidges Tedlar Bags	N/A	14 days until extraction; Tedlar Bags – transfer onto sorbent tube within 72 hours. 30 days for analysis	30L Cartridges Bags 500ml

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Holding Time	Sample Vol.[c]
Methanol, Acetaldehyde, Methyl Ethyl Ketone, Propionaldehyde (NCASI – DI/MeOH 94.03 /NCASI – DI/HAPS 99.01)	Aqueous - Effluent	Glass w/Teflon Lined Lid	No Headspace; 4˚C±2˚C; HCl to pH 2-3 (Effluent only)	30 days	(1) 40mL Vial
Reduced Sulfur Compounds (NCASI Method RSC-02.02)	Aqueous	40ml amber, borosilicate glass vials with Teflon faced silicone backed caps.	MeSH, DMS, and DMTS (RSCs non-H2S) addition of ascorbic acid and pH adjustment to <2.5 with 1:2 phosphoric acid solution upon collection. Laboratory Preservation, 4˚C±2	14 days	(2) 40ml VOA Vials
Total Sulfide (NCASI Method RSC-02.02)	Aqueous	40ml amber, borosilicate glass vials with Teflon faced silicone backed caps.	Addition of Zinc acetate solution and pH adjustment to >10 with 1 N NaOH solution upon collection. Laboratory Preservation, 4˚C±2	14 days	(2) 40ml VOA Vials
Hydrofluoric Acid (In-House Method)	Air	Radiello Samplers	Laboratory Preservation, 4˚C±2	4 months	15 minutes to 14 days exposure (dependent on sampling environment)
Hydrogen Sulfide (In-House Method)	Air	Radiello Samplers	N/A	6 months	1 hour to 15 days exposure (dependent on sampling environment)
Nitrogen Dioxide (In-House Method)	Air	Radiello samplers	Laboratory Preservation, store in dark at 4˚C±2	4 months	7 to 15 days exposure (dependent on sampling environment)

Sample Preservation and Holding Times for Performed Methods

Determination (Method)	Matrix	Container	Preservation	Holding Time	Sample Vol.[c]
Ozone (In-House Method)	Air	Radiello Samplers	Protect from light	7 days	24 hours to 14 days exposure (dependent on sampling environment)
Sulfur Dioxide (In-House Method)	Air	Radiello Samplers	Laboratory Preservation, store in dark at 4˚C±2	4 months	7 to 15 days exposure (dependent on sampling environment)

Footnotes:

a.	Some methods do not specify the utilization of canisters; therefore, there is no required hold time and this will be noted in the case narrative.
b.	Laboratory recommended hold time; therefore, samples analyzed outside this hold time will be noted in the case narrative accordingly.
c.	Sample volumes are the minimum, which should be received by the laboratory; however, canister volumes should match the canister size utilized.
d.	There is no holding time requirement available and laboratory studies are not available indicating the validity of data prior to or following a specified length of time. Therefore, no holding time notation or qualifier will be adhered to results.

APPENDIX G – Standard Operating Procedures

Corporate SOP Titles	SOP ID
Laboratory Ethics and Data Integrity	CE-GEN001
(Proprietary – Client Specific)	CE-GEN002
Records Management Policy	CE-GEN003
Preventive Action	CE-GEN004
Document Control	CE-GEN005
Data Recall	CE-GEN006
Procurement and Control of Laboratory Services and Supplies	CE-GEN007
Method Development	CE-GEN008
Establishing Standard Operating Procedures	CE-GEN009
Handling Customer Feedback	CE-GEN010
Assigning a TSR to a Project	CE-GEN011
Policy for the use of Accreditation Organization Names, Symbols, and Logos	CE-GEN012
(Proprietary – Client Specific)	CE-GEN013
Internal Audits	CE-QA001
Manual Integration Policy	CE-QA002
Training Policy	CE-QA003
Qualification of Subcontract Laboratories	CE-QA004
Laboratory Management Review	CE-QA005
Proficiency Testing	CE-QA006
Making Entries onto Analytical Records	CE-QA007
Nonconformance and Corrective Action	CE-QA008
Control Limits	CE-QA009
Estimation of Uncertainty of Analytical Measurements	CE-QA010
Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	CE-QA011
Quality of Reagents and Standards	CE-QA012

Local Administrative SOP Titles	SOP Code
Data and Record Archiving	ADM-ARC
Batches and Sequences	ADM-BATCH_SEQ
Handling Consumable Materials	ADM-CONSUM
Electronic Data Backup, Archiving, and Restoration	ADM-DATA_BU
Data Review and Reporting	ADM-DATA_REV
Glassware Cleaning	ADM-GLASS
Analytical Instrument Acquisition, Reassignment, Maintenance and Documentation	ADM-INSTRUM
Laboratory Storage, Analysis, and Tracking	ADM-LabSAT
Media Request Fulfillment	ADM-Media_Req
Project Management	ADM-PMgmt
Software and Data Quality Assurance	ADM-SftwreQA
Significant Figures	ADM-SIG_FIG
Calibration and Use of Laboratory Support Equipment	ADM-SupEQ
Waste Disposal	ADM-WASTE
Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters	SMO-Can_Cert
Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters	SMO-Can_Press
Flow Controllers and Critical Orifices	SMO-Flow_Cntrl
Sample Receiving, Acceptance and Log-In	SMO-SMPL_REC

Semi-Volatile SOP Titles	SOP Code
Determination of Formaldehyde and Other Carbonyl Compounds in Ambient Air Using Adsorbent Cartridge Followed by High Performance Liquid Chromatography (HPLC) EPA Compendium Method T0-11A	SVO-11A
Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS)	SVO-13A
Determination of Volatile Amines in Ambient Air Using Gas Chromatography Equipped with a Nitrogen Phosphorus Detector (NPD)	SVO-AMINES
Determination of Carboxylic Acids in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS)	SVO-CACIDS
Analysis of Halogenated Volatile Organic Compounds in Emissions from Stationary Sources using GC/ECD in Accordance with a Modification of CARB Method 422	SVO-CARB422
NCASI Method RSC-02.02 Reduced Sulfur Compounds by Direct Injection GC/PFPD	SVO-NCASI_RSC
Determination of Methanol, Acetaldehyde, Methyl Ethyl Ketone, and Propionaldehyde in Pulp and Paper Process Liquids by GC/FID	SVO-NCASI_MeOH
Preparation and Analysis of 2-Butoxyethanol on Coconut Shell Charcoal Tubes and Analyzed using GC/FID	SVO-NIOSH1403
Determination of Organic Vapors Using GC/FID in Accordance with OSHA Method 07	SVO-OSHA_07
Determination of P-9290 Target Compounds from a Chamber and Specific P-9290 Quality Control Parameters	SVO-P9290
Preparation and Analysis of Orthorhombic Cyclooctasulfur by Gas Chromatography/Electron Capture Detector (GC/ECD)	SVO-S8_ECD
Analysis of Sulfur Hexafluoride in Accordance with a Modification of NIOSH 6602	SVO-SF6
Determination of Siloxanes in Biogas using Gas Chromatography/Mass Spectrometry (GC/MS)	SVO-SILOXANES
Determination of Pesticides and Polychlorinated Biphenyls (PCBs) in Ambient Air by GC/ECD per EPA Compendium Methods TO-4 and TO-10A	SVO-TO4A
Sample and Media Preparation per EPA Compendium Method TO-13A	SVP-TO13A
Sample Extraction and Preparation of Pesticide and PCB Samples According to EPA Compendium Methods TO-4A and TO-10A	SVP-TO4A

Volatile SOP Titles	SOP Code
Analysis of Air Corrosivity by Checkmate Meter	VOA-AIRCORR
Analysis of Argon Using Gas Chromatography with Thermal Conductivity Detection (TCD)	VOA-ARGON
Calculating Heat Value, Compressibility Factor, and Relative Density of Gaseous Fuels in Accordance with ASTM D 3588	VOA-BTU
Samples Preparation in Glass Chambers	VOA-CHAMBER
Dissolved Gas Analysis in Aqueous Samples Using a GC Headspace Equilibration Technique	VOA-DISGAS
Sample Preparation of Drywall for Sulfur Analysis and the Determination of Copper Corrosion	VOA-DRYWALL
Determination of Total Gaseous Nonmethane Organic (TGNMO) Emissions as Carbon in Landfill Gases in Accordance with EPA Method 25C	VOA-EPA25C
Determination of Methane, Carbon Monoxide, Carbon Dioxide, and Total Gaseous Nonmethane Organic (TGNMO) Emissions as Carbon in Landfill Gases According to Modified EPA Method 25C	VOA-EPA25CM
Determination of Hydrogen, Carbon Monoxide, Carbon Dioxide, Nitrogen, Methane, and Oxygen using Gas Chromatography with Thermal Conductivity Detection (TCD) in Accordance with EPA 3C or ASTM D 1946	VOA-EPA3C
Analysis of Hydrogen and Helium using Gas Chromatography with Thermal Conductivity Detection (TCD)	VOA-HHe
Analysis of Sulfur Compounds in a Gaseous Matrix by Gas Chromatography with Sulfur Chemiluminescence Detection per ASTM D 5504 and Modified SCAQMD Method 307	VOA-S307M_SCD
Analysis of Sulfur Compounds in Liquid Samples by Gas Chromatography with Sulfur Chemiluminescence Detection	VOA-SH_2O_SCD
Analysis of C1-C6+ using Gas Chromatography with Flame Ionization Detection (FID) in Accordance with a Modification of EPA Compendium Method TO-3	VOA-TO3C1C6
Analysis of Various Compounds using Gas Chromatography with Flame Ionization Detection (FID) in Accordance with a Modification of EPA Compendium Method TO-3	VOA-TO3MeOH
Analysis of Total Petroleum Hydrocarbons as Gasoline in Air by Gas Chromatography with Flame Ionization Detection	VOA-TPHG_TO3
Determination of Air-Phase Petroleum Hydrocarbons by Gas Chromatography/Mass Spectrometry (GC/MS)	VOA-MAPH
Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)	VOA-TO15
Determination of Volatile Organic Compounds in Ambient Air Using Active or Passive Sampling Onto Sorbent Tubes	VOA-TO17

General Chemistry (WET) SOP Titles	SOP Code
Determination of Inorganic Anions by Ion Chromatography	WET-Anions_IC
Colorimetric Determination of Hydrogen Sulfide (H_2S) in Air	WET-H_2SAir
Analysis of Hydrofluoric (HF) Acid in Air by Ion Selective Electrode	WET-HFAir
Ammonia in Air by Ion Selective Electrode	WET-NH_3Air
Colorimetric Determination of Nitrogen Dioxide (NO_2) in Air	WET-NO_2Air
Colorimetric Determination of Ozone (O_3) in Air	WET-O_3Air
pH Electrometric Measurement for Liquids by Ion Selective Electrodes	WET-pHL
pH Electrometric Measurement for Solids by Ion Selective Electrodes	WET-pHS

APPENDIX H – Data Qualifiers

CODE	CATEGORY	DESCRIPTION
BC	AIHA	Reported results are not blank corrected.
BH	AIHA	Results indicate breakthrough; back section of tube greater than front section.
BT	AIHA	Results indicated possible breakthrough; back section ≥10% front section.
DE	AIHA	Reported results are corrected for desorption efficiency.
RA	AIHA	Result not available.
G	GENERAL	Improper container.
G1	GENERAL	Unpreserved or improperly preserved sample.
X	GENERAL	See case narrative.
H1	HOLD TIME	Sample analysis performed past holding time. See case narrative.
H2	HOLD TIME	Initial analysis within holding time. Reanalysis for the required dilution was past holding time.
H3	HOLD TIME	Sample was received and analyzed past holding time.
H4	HOLD TIME	Sample was extracted past required extraction holding time, but analyzed within analysis holding time. See case narrative.
i	MATRIX	The MDL/MRL has been elevated due to matrix interference.
M	MATRIX	Matrix interference; results may be biased (high/low).
M1	MATRIX	Matrix interference due to coelution with a non-target compound. (TO-15 only)
Q	PETROLEUM	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of lighter/heavier molecular weight constituents than the calibration standard.
Y	PETROLEUM	The chromatogram resembles a petroleum product but does not match the calibration standard.
Z	PETROLEUM	The chromatogram does not resemble a petroleum product.
#	QC	The control limit criterion is not applicable. See case narrative.
*	QC	The result is an outlier. See case narrative.
B	QC	Analyte detected in both the sample and associated method blank.
I	QC	Internal standard not within the specified limits. See case narrative.
L	QC	Laboratory control sample recovery outside the specified limits; results may be biased (high/low).
N	QC	The matrix spike sample recovery is not within control limits. See case narrative.
R	QC	Duplicate precision not met.

CODE	CATEGORY	DESCRIPTION
R1	QC	Duplicate precision not within the specified limits; however, the results are below the MRL and considered estimated.
S	QC	Surrogate recovery not within specified limits.
V	QC	The continuing calibration verification standard was outside (biased high/low) the specified limits for this compound.
C	RESULT	Result identification confirmed.
CE	RESULT	Co-elution.
D	RESULT	The reported result is from a dilution.
E	RESULT	Estimated; concentration exceeded calibration range.
J	RESULT	The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
J1	RESULT	The analyte was positively identified below the method reporting limit prior to utilizing the dilution factor; the associated numerical value is considered estimated.
K	RESULT	Analyte was detected above the method reporting limit prior to normalization.
ND	RESULT	Compound was analyzed for, but not detected above the laboratory reporting/detection limit.
P	RESULT	The confirmation criterion was exceeded. The relative percent difference was greater than 40/25% between the two analytical results.
U	RESULT	Compound was analyzed for, but not detected (ND) at or above the MRL/MDL.
W	RESULT	Result quantified, but the corresponding peak was detected outside the generated retention time window.
UJ	RESULT	The analyte was not detected; however, the result is estimated due to discrepancies in meeting certain analyte-specific quality control criteria.
Ui	RESULT	The compound was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL; however, the MRL/MDL has been elevated due to matrix interference.
T	TIC	Analyte is a tentatively identified compound, result is estimated.

APPENDIX I – Master List of Controlled Documents

Controlled Documents*	Document Code
Health and Safety Manual	ADM-SAFETY
Quality Assurance Manual	ALSMV-QAM

*Refer to Appendix G for a list of the laboratory's controlled standard operating procedures.

QA Program Files	
Item	**Location / Name**
Approved Signatories List	QA Manual Appendix I
Approved Subcontract Laboratories	Q:\Approved Sub-Contract Labs\Subcontract Lab List
Control Limit\Chart Status	Q:\Control Charts\CntrlChrt(status1).xls
Job Descriptions	HR Department
Master List of Controlled Documents (Logbooks, SOPs, etc.)	Q:\Master List of Controlled Documents\Master List of Controlled Documents.xls
MDL,LOD,LOQ Status	Q:\MDL Status\MDL Status Table (EACH DEPT).xls
Personnel Resumes, Transcripts	HR and QA Departments
Simi Valley Certification Status	Q:\Certifications\Cert Status.xls
Simi Valley Data Quality Objectives	Q:\MDL_MRL\DQO Spreadsheet.xls
Technical Training Status	Q:\Training\TRAINING STATUS\TRAINING STATUS.xls

Approved Signatories	
Name	**Title**
Kelly Horiuchi, B.A.	Laboratory Director / Project Manager
Chaney Humphrey, B.S.	Quality Assurance Manager
Wade Henton, B.S.	Volatiles (GC) Technical Manager
Chris Parnell, B.S.	Operations Manager; Technical Manager (VOA GC/MS – Air)
Madeleine Dangazyan, B.S.	Semi-Volatiles/ Industrial Hygiene Technical Manager; Environmental Health & Safety Coordinator
Wida Ang, B.S., M.S.	Team Leader (Volatiles GC/MS – Air)
Sue Anderson, B.S.	Project Manager / Technical Manager (General Chemistry)
Samantha Henningsen, B.S.	Project Manager
Kathleen Aguilera, B.A.	Client Services Manager / Project Manager

APPENDIX J – Laboratory Accreditations

American Industrial Hygiene Association (AIHA)

Industrial Hygiene Laboratory Accreditation Program Laboratory
Laboratory # 101661
Approved Method(s):

- NIOSH 1450
- NIOSH 1457
- NIOSH 1500
- NIOSH 1501
- NIOSH 1550
- OSHA 07

State of Arizona, Department of Health Services

License No. AZ0694
Approved Method(s):

- EPA TO-15
- EPA 3C

Department of Defense, Environmental Laboratory Accreditation Program (DoD-ELAP)

Perry Johnson Laboratory Accreditation, Inc. Accreditation No. 65818
Approved Method(s):

- EPA TO-15
- RSK 175
- EPA 3C
- ASTM D 1946-90
- SOP VOA-EPA3C (EPA 3C Modified)
- SOP VOA-TPHG_TO3 (TPHG by Modified EPA TO-3)
- SOP VOA-TO3C1C6 (Hydrocarbons and ranges by Modified EPA TO-3)
- SOP VOA-TO15 (EPA TO-15 Modified)

State of Florida, Department of Health (NELAP-Secondary)

Laboratory ID No.: E871020
Approved Method(s):

- EPA TO-15
- EPA TO-17

State of Maine, Department of Health and Human Services

Certificate No.: 2014025
Approved Methods

- EPA TO-15
- MADEP APH

State of Minnesota, Department of Health, Environmental Laboratory Certification Program (NELAP-Secondary)
Laboratory ID: 006-999-456
Approved Method(s):
- EPA TO-15

State of New York, Department of Health (NELAP –Secondary)
Environmental Analyses/Air and Emissions
Laboratory ID No. 11221
Approved Method(s):
- EPA TO-13A
- EPA TO-15
- EPA TO-17

State of New Jersey, Department of Environmental Protection (NELAP-Secondary)
Laboratory ID: CA009
Approved Method(s):
- EPA TO-15
- EPA TO-13A

State of Oregon, Environmental Laboratory Accreditation Program (NELAP-Primary)
Laboratory ID: 4068
Approved Method(s):
- EPA TO-4A
- EPA TO-10A
- EPA TO13A
- EPA TO-15
- EPA TO-17
- MADEP APH

Commonwealth of Pennsylvania, Department of Environmental Protection Bureau of Laboratories
Registration Number: 68-03307

State of Texas, Texas Commission on Environmental Quality (NELAP-Secondary)
Certificate # T104704413-14-5
Approved Method(s):
- EPA TO-15

State of Utah, Department of Health, Environmental Laboratory Certification Program (NELAP-Secondary)
Certificate # CA016272014-4
Approved Method(s):
- EPA TO-15

State of Washington, Department of Ecology
Laboratory ID: C946
Approved Method(s):
- EPA TO-15
- EPA RSK-175

Note 1: This Quality Assurance Manual is revised annually with AIHA, DoD and NELAP-Primary Certificates, and the Scope of Accreditations/Parameters are revised annually (where necessary). During this interim period Certificates may expire and the Scope of Accreditations/Parameters may change; therefore, these may not be updated until the next revision.

Note 2: Current Certificates and Scope of Accreditations/Parameters are on file and displayed in the front hallway. Updated or Specific Certificates and Scope of Accreditations/Parameters are available upon request.

AIHA Laboratory Accreditation Programs, LLC

acknowledges that

ALS Environmental – Simi Valley
2655 Park Center Drive, Suite A, Simi Valley, CA 93065-6200
Laboratory ID: 101661

along with all premises from which key activities are performed, as listed above, has fulfilled the requirements of the AIHA Laboratory Accreditation Programs (AIHA-LAP), LLC accreditation to the ISO/IEC 17025:2005 international standard, *General Requirements for the Competence of Testing and Calibration Laboratories* in the following:

LABORATORY ACCREDITATION PROGRAMS

✓	**INDUSTRIAL HYGIENE**	Accreditation Expires: 11/01/2016
☐	**ENVIRONMENTAL LEAD**	Accreditation Expires:
☐	**ENVIRONMENTAL MICROBIOLOGY**	Accreditation Expires:
☐	**FOOD**	Accreditation Expires:
☐	**UNIQUE SCOPES**	Accreditation Expires:

Specific Field(s) of Testing (FoT)/Method(s) within each Accred
itation Program for which the above named laboratory maintains accreditation is outlined on the attached **Scope of Accreditation**. Continued accreditation is contingent upon successful on-going compliance with ISO/IEC 17025:2005 and AIHA-LAP, LLC requirements. This certificate is not valid without the attached **Scope of Accreditation**. Please review the AIHA-LAP, LLC website (www.aihaaccreditedlabs.org) for the most current Scope.

Gerald Schultz, CIH
Chairperson, Analytical Accreditation Board

Cheryl O. Morton
Managing Director, AIHA Laboratory Accreditation Programs, LLC

Revision 14: 03/26/2014

Date Issued: 09/30/2014

AIHA Laboratory Accreditation Programs, LLC
SCOPE OF ACCREDITATION

ALS Environmental – Simi Valley
2655 Park Center Drive Suite A, Simi Valley, CA 93065-6200

Laboratory ID: **101661**
Issue Date: 09/30/2014

The laboratory is approved for those specific field(s) of testing/methods listed in the table below. Clients are urged to verify the laboratory's current accreditation status for the particular field(s) of testing/Methods, since these can change due to proficiency status, suspension and/or withdrawal of accreditation.

Industrial Hygiene Laboratory Accreditation Program (IHLAP)

Initial Accreditation Date: 09/01/1994

IHLAP Scope Category	Field of Testing (FoT)	Technology sub-type/ Detector	Published Reference Method/Title of In-house Method	Method Description or Analyte *(for internal methods only)*
Chromatography Core	Gas Chromatography	GC/FID	NIOSH 1450	
			NIOSH 1457	
			NIOSH 1500	
			NIOSH 1501	
			NIOSH 1550	
			OSHA 07	
	Gas Chromatography (Diffusive Samplers)		OSHA 07	

A complete listing of currently accredited Industrial Hygiene laboratories is available on the AIHA-LAP, LLC website at: http://www.aihaaccreditedlabs.org

Effective: 03/12/2013
101661_Scope_IHLAP_2014_09_30.docx
Page 1 of 1

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

ALS Environmental
2655 Park Center Drive, Suite A, Simi Valley, CA 93065

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the DoD Quality Systems Manual for Environmental Laboratories Version 4.2 10/26/2010 and is accredited is accordance with the:

United States Department of Defense
Environmental Laboratory Accreditation Program
(DoD-ELAP)

This accreditation demonstrates technical competence for the defined scope:
Environmental Testing
(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body's duty to observe and comply with the said rules.

For PJLA:

Tracy Szerszen
President/Operations Manager

Initial Accreditation Date:	*Issue Date:*	*Expiration Date:*
January 11, 2010	January 2, 2014	January 31, 2016

Accreditation No.:	*Certificate No.:*
65818	L14-2

Perry Johnson Laboratory
Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

Page 1 of 8

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Aqueous	RSK 175	GC/FID	Methane
Aqueous	RSK 175	GC/FID	Ethane
Aqueous	RSK 175	GC/FID	Ethene
Aqueous	RSK 175	GC/TCD	Carbon Dioxide
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Hydrogen
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Oxygen
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Nitrogen
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Methane
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Carbon Dioxide
Air	(ALS SOP) VOA-EPA3C	GC/TCD	Carbon Monoxide
Air	(ALS SOP) VOA-TPHG_TO3	GC/FID	Total Petroleum Hydrocarbons Gasoline (TPHG)
Air	(ALS SOP) VOA-TPHG_TO3	GC/FID	JP-4
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	C1 - C6+
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Ethane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Ethene
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Methane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	n-Butane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	n-Hexane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	n-Pentane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Propane
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Propene
Air	(ALS SOP) VOA-TO3C1C6	GC/FID	Total Volatile Petroleum Hydrocarbons (TVPH) as Hexane
Air	EPA TO-15	GC/MS	1,1,1-Trichloroethane
Air	EPA TO-15	GC/MS	1,1,2,2-Tetrachloroethane
Air	EPA TO-15	GC/MS	1,1,2-Trichloroethane
Air	EPA TO-15	GC/MS	1,1-Dichloroethane
Air	EPA TO-15	GC/MS	1,1-Dichloroethene
Air	EPA TO-15	GC/MS	1,2,3-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,2,4-Trichlorobenzene

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	1,2,4-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,2,Dibromo-3-Chloropropane
Air	EPA TO-15	GC/MS	1,2-Dibromoethane
Air	EPA TO-15	GC/MS	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
Air	EPA TO-15	GC/MS	1,2-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,2-Dichloroethane
Air	EPA TO-15	GC/MS	1,2-Dichloropropane
Air	EPA TO-15	GC/MS	1,3,5-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,3-Butadiene
Air	EPA TO-15	GC/MS	1,3-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,4-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,4-Dioxane
Air	EPA TO-15	GC/MS	1-Butanol
Air	EPA TO-15	GC/MS	2-Butanone (MEK)
Air	EPA TO-15	GC/MS	2-Ethyltoluene
Air	EPA TO-15	GC/MS	2-Hexanone
Air	EPA TO-15	GC/MS	3-Ethyltoluene
Air	EPA TO-15	GC/MS	4-Ethyltoluene
Air	EPA TO-15	GC/MS	4-Methyl-2-Pentanone
Air	EPA TO-15	GC/MS	Acetone
Air	EPA TO-15	GC/MS	Acetonitrile
Air	EPA TO-15	GC/MS	Acrolein
Air	EPA TO-15	GC/MS	Acrylonitrile
Air	EPA TO-15	GC/MS	Allyl Chloride
Air	EPA TO-15	GC/MS	alpha-Methylstyrene
Air	EPA TO-15	GC/MS	alpha-Pinene
Air	EPA TO-15	GC/MS	Benzene
Air	EPA TO-15	GC/MS	Benzyl Chloride
Air	EPA TO-15	GC/MS	Bromodichloromethane
Air	EPA TO-15	GC/MS	Bromoform
Air	EPA TO-15	GC/MS	Bromomethane
Air	EPA TO-15	GC/MS	Carbon Disulfide
Air	EPA TO-15	GC/MS	Carbon Tetrachloride
Air	EPA TO-15	GC/MS	Chlorobenzene
Air	EPA TO-15	GC/MS	Chloroethane

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 3 of 8*

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	Chloroform
Air	EPA TO-15	GC/MS	Chloromethane
Air	EPA TO-15	GC/MS	cis-1,2-Dichloroethene
Air	EPA TO-15	GC/MS	cis-1,3-Dichloropropene
Air	EPA TO-15	GC/MS	Cumene
Air	EPA TO-15	GC/MS	Cyclohexane
Air	EPA TO-15	GC/MS	Cyclohexanone
Air	EPA TO-15	GC/MS	Dibromochloromethane
Air	EPA TO-15	GC/MS	Dichlorodifluoromethane (CFC 12)
Air	EPA TO-15	GC/MS	Diisopropyl Ether
Air	EPA TO-15	GC/MS	d-Limonene
Air	EPA TO-15	GC/MS	Ethanol
Air	EPA TO-15	GC/MS	Ethyl Acetate
Air	EPA TO-15	GC/MS	Ethyl tert-Butyl Ether
Air	EPA TO-15	GC/MS	Ethylbenzene
Air	EPA TO-15	GC/MS	Hexachlorobutadiene
Air	EPA TO-15	GC/MS	Isooctane
Air	EPA TO-15	GC/MS	Isopropyl acetate
Air	EPA TO-15	GC/MS	Isopropyl Alcohol
Air	EPA TO-15	GC/MS	m-&, p-Xylenes
Air	EPA TO-15	GC/MS	Methyl Methacrylate
Air	EPA TO-15	GC/MS	Methyl tert-Butyl Ether
Air	EPA TO-15	GC/MS	Methylene Chloride
Air	EPA TO-15	GC/MS	Naphthalene
Air	EPA TO-15	GC/MS	n-Butyl Acetate
Air	EPA TO-15	GC/MS	n-Butylbenzene
Air	EPA TO-15	GC/MS	n-Decane
Air	EPA TO-15	GC/MS	n-Dodecane
Air	EPA TO-15	GC/MS	n-Heptane
Air	EPA TO-15	GC/MS	n-Hexane
Air	EPA TO-15	GC/MS	n-Nonane

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	n-Octane
Air	EPA TO-15	GC/MS	n-Propylbenzene
Air	EPA TO-15	GC/MS	n-Undecane
Air	EPA TO-15	GC/MS	o-Xylene
Air	EPA TO-15	GC/MS	p-Isopropyltoluene
Air	EPA TO-15	GC/MS	Propene
Air	EPA TO-15	GC/MS	sec-Butylbenzene
Air	EPA TO-15	GC/MS	Styrene
Air	EPA TO-15	GC/MS	tert-Amyl Methyl Ether
Air	EPA TO-15	GC/MS	tert-Butanol
Air	EPA TO-15	GC/MS	tert-Butylbenzene
Air	EPA TO-15	GC/MS	Tetrachloroethene
Air	EPA TO-15	GC/MS	Tetrahydrofuran
Air	EPA TO-15	GC/MS	Toluene
Air	EPA TO-15	GC/MS	trans-1,2-Dichloroethene
Air	EPA TO-15	GC/MS	trans-1,3-Dichloropropene
Air	EPA TO-15	GC/MS	Trichloroethene
Air	EPA TO-15	GC/MS	Trichlorofluoromethane
Air	EPA TO-15	GC/MS	Trichlorotrifluoroethane
Air	EPA TO-15	GC/MS	Vinyl Acetate
Air	EPA TO-15	GC/MS	Vinyl Chloride
Air	ASTM D 1946-90	GC/TCD	Hydrogen
Air	ASTM D 1946-90	GC/TCD	Oxygen
Air	ASTM D 1946-90	GC/TCD	Nitrogen
Air	ASTM D 1946-90	GC/TCD	Methane
Air	ASTM D 1946-90	GC/TCD	Carbon Dioxide
Air	ASTM D 1946-90	GC/TCD	Carbon Monoxide
Air	EPA 3C	GC/TCD	Oxygen
Air	EPA 3C	GC/TCD	Nitrogen
Air	EPA 3C	GC/TCD	Methane
Air	EPA 3C	GC/TCD	Carbon Dioxide
Air	(ALS SOP) VOA-TO15	GC/MS	1,1,1-Trichloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,1,2,2-Tetrachloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,1,2-Trichloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,1-Dichloroethane

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 5 of 8*

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	(ALS SOP) VOA-TO15	GC/MS	1,1-Dichloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2,3-Trimethylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2,4-Trichlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2,4-Trimethylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dibromo-3-Chloropropane
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dibromoethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dichlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dichloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	1,2-Dichloropropane
Air	(ALS SOP) VOA-TO15	GC/MS	1,3,5-Trimethylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,3-Butadiene
Air	(ALS SOP) VOA-TO15	GC/MS	1,3-Dichlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,4-Dichlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	1,4-Dioxane
Air	(ALS SOP) VOA-TO15	GC/MS	1-Butanol
Air	(ALS SOP) VOA-TO15	GC/MS	2-Butanone (MEK)
Air	(ALS SOP) VOA-TO15	GC/MS	2-Ethyltoluene
Air	(ALS SOP) VOA-TO15	GC/MS	2-Hexanone
Air	(ALS SOP) VOA-TO15	GC/MS	3-Ethyltoluene
Air	(ALS SOP) VOA-TO15	GC/MS	4-Ethyltoluene
Air	(ALS SOP) VOA-TO15	GC/MS	4-Methyl-2-Pentanone
Air	(ALS SOP) VOA-TO15	GC/MS	Acetone
Air	(ALS SOP) VOA-TO15	GC/MS	Acetonitrile
Air	(ALS SOP) VOA-TO15	GC/MS	Acrolein
Air	(ALS SOP) VOA-TO15	GC/MS	Acrylonitrile
Air	(ALS SOP) VOA-TO15	GC/MS	Allyl Chloride
Air	(ALS SOP) VOA-TO15	GC/MS	alpha-Methylstyrene
Air	(ALS SOP) VOA-TO15	GC/MS	alpha-Pinene
Air	(ALS SOP) VOA-TO15	GC/MS	Benzene
Air	(ALS SOP) VOA-TO15	GC/MS	Benzyl Chloride
Air	(ALS SOP) VOA-TO15	GC/MS	Bromodichloromethane
Air	(ALS SOP) VOA-TO15	GC/MS	Bromoform
Air	(ALS SOP) VOA-TO15	GC/MS	Bromomethane

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 6 of 8*

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	(ALS SOP) VOA-TO15	GC/MS	Carbon Disulfide
Air	(ALS SOP) VOA-TO15	GC/MS	Carbon Tetrachloride
Air	(ALS SOP) VOA-TO15	GC/MS	Chlorobenzene
Air	(ALS SOP) VOA-TO15	GC/MS	Chloroethane
Air	(ALS SOP) VOA-TO15	GC/MS	Chloroform
Air	(ALS SOP) VOA-TO15	GC/MS	Chloromethane
Air	(ALS SOP) VOA-TO15	GC/MS	cis-1,2-Dichloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	cis-1,3-Dichloropropene
Air	(ALS SOP) VOA-TO15	GC/MS	Cumene
Air	(ALS SOP) VOA-TO15	GC/MS	Cyclohexane
Air	(ALS SOP) VOA-TO15	GC/MS	Cyclohexanone
Air	(ALS SOP) VOA-TO15	GC/MS	Dibromochloromethane
Air	(ALS SOP) VOA-TO15	GC/MS	Dichlorodifluoromethane (CFC 12)
Air	(ALS SOP) VOA-TO15	GC/MS	Diisopropyl Ether
Air	(ALS SOP) VOA-TO15	GC/MS	d-Limonene
Air	(ALS SOP) VOA-TO15	GC/MS	Ethanol
Air	(ALS SOP) VOA-TO15	GC/MS	Ethyl Acetate
Air	(ALS SOP) VOA-TO15	GC/MS	Ethyl tert-Butyl Ether
Air	(ALS SOP) VOA-TO15	GC/MS	Ethylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	Hexachlorobutadiene
Air	(ALS SOP) VOA-TO15	GC/MS	Isooctane
Air	(ALS SOP) VOA-TO15	GC/MS	Isopropyl acetate
Air	(ALS SOP) VOA-TO15	GC/MS	Isopropyl Alcohol
Air	(ALS SOP) VOA-TO15	GC/MS	m-&,p-Xylenes
Air	(ALS SOP) VOA-TO15	GC/MS	Methyl Methacrylate
Air	(ALS SOP) VOA-TO15	GC/MS	Methyl tert-Butyl Ether
Air	(ALS SOP) VOA-TO15	GC/MS	Methylene Chloride
Air	(ALS SOP) VOA-TO15	GC/MS	Naphthalene
Air	(ALS SOP) VOA-TO15	GC/MS	n-Butyl Acetate
Air	(ALS SOP) VOA-TO15	GC/MS	n-Butylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	n-Decane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Dodecane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Heptane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Hexane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Nonane

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 7 of 8*

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard Method	Technology	Analyte
Air	(ALS SOP) VOA-TO15	GC/MS	n-Octane
Air	(ALS SOP) VOA-TO15	GC/MS	n-Propylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	n-Undecane
Air	(ALS SOP) VOA-TO15	GC/MS	o-Xylene
Air	(ALS SOP) VOA-TO15	GC/MS	p-Isopropyltoluene
Air	(ALS SOP) VOA-TO15	GC/MS	Propene
Air	(ALS SOP) VOA-TO15	GC/MS	sec-Butylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	Styrene
Air	(ALS SOP) VOA-TO15	GC/MS	tert-Amyl Methyl Ether
Air	(ALS SOP) VOA-TO15	GC/MS	t-Butanol
Air	(ALS SOP) VOA-TO15	GC/MS	tert-Butylbenzene
Air	(ALS SOP) VOA-TO15	GC/MS	Tetrachloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	Tetrahydrofuran
Air	(ALS SOP) VOA-TO15	GC/MS	Toluene
Air	(ALS SOP) VOA-TO15	GC/MS	trans-1,2-Dichloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	trans-1,3-Dichloropropene
Air	(ALS SOP) VOA-TO15	GC/MS	Trichloroethene
Air	(ALS SOP) VOA-TO15	GC/MS	Trichlorofluoromethane
Air	(ALS SOP) VOA-TO15	GC/MS	Trichlorotrifluoroethane
Air	(ALS SOP) VOA-TO15	GC/MS	Vinyl Acetate
Air	(ALS SOP) VOA-TO15	GC/MS	Vinyl Chloride

Issue: 1/14 *This supplement is in conjunction with certificate #L14-2* *Page 8 of 8*

OREGON

Environmental Laboratory Accreditation Program

NELAP Recognized

ALS Environmental - Simi Valley

4068

2655 Park Center Drive, Suite A

Simi Valley,CA 93065

IS GRANTED APPROVAL BY ORELAP UNDER THE 2009 TNI STANDARDS, TO PERFORM ANALYSES ON ENVIRONMENTAL SAMPLES IN MATRICES AS LISTED BELOW :

Air	*Drinking Water*	*Non Potable Water*	*Solids and Chem. Waste*	*Tissue*
Chemistry		Chemistry		

AND AS RECORDED IN THE LIST OF APPROVED ANALYTES, METHODS, ANALYTICAL TECHNIQUES, AND FIELDS OF TESTING ISSUED CONCURRENTLY WITH THIS CERTIFICATE AND REVISED AS NECESSARY.

ACCREDITED STATUS DEPENDS ON SUCCESSFUL ONGOING PARTICIPATION IN THE PROGRAM AND CONTINUED COMPLIANCE WITH THE STANDARDS.

CUSTOMERS ARE URGED TO VERIFY THE LABORATORY'S CURRENT ACCREDITATION STATUS IN OREGON.

Gary K. Ward, MS
Oregon State Public Health Laboratory
ORELAP Administrator
3150 NW. 229th Ave, Suite 100
Hillsboro, OR 97124

ISSUE DATE: 02/16/2015
EXPIRATION DATE: 02/15/2016
Certificate No: 4068 - 001

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Oregon

Environmental Laboratory Accreditation Program

Department of Agriculture, Laboratory Division
Department of Environmental Quality, Laboratory Division
Oregon Health Authority, Public Health Division

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ORELAP Fields of Accreditation

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MATRIX : Air

Reference	Code	Description
EPA TO-10A (GC/ECD)	**10247504**	**Pesticides and PCBs with LV PUF by GC/ECD**

Analyte Code	*Analyte*
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
8880	Aroclor-1016 (PCB-1016)
8910	Aroclor-1260 (PCB-1260)
7115	beta-BHC (beta-Hexachlorocyclohexane)
7105	delta-BHC
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide
7810	Methoxychlor

EPA TO-13A	**10248405**	**Polycyclic Aromatic Hydrocarbons in Ambient Air by GC/MS**

Analyte Code	*Analyte*
5500	Acenaphthene
5505	Acenaphthylene
5555	Anthracene
5575	Benzo(a)anthracene
5580	Benzo(a)pyrene
5590	Benzo(g,h,i)perylene
5600	Benzo(k)fluoranthene
5585	Benzo[b]fluoranthene
5855	Chrysene
5895	Dibenz(a,h) anthracene
6265	Fluoranthene
6270	Fluorene
6315	Indeno(1,2,3-cd) pyrene

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Analyte Code	*Analyte*
5005	Naphthalene
6615	Phenanthrene
6665	Pyrene

EPA TO-15 **10248803** **VOCs collected in Canisters by GC/MS**

Analyte Code	*Analyte*
5160	1,1,1-Trichloroethane
5110	1,1,2,2-Tetrachloroethane
5195	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
5165	1,1,2-Trichloroethane
4630	1,1-Dichloroethane
4640	1,1-Dichloroethylene
5182	1,2,3-Trimethylbenzene
5155	1,2,4-Trichlorobenzene
5210	1,2,4-Trimethylbenzene
4570	1,2-Dibromo-3-chloropropane (DBCP)
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
4695	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane (Ethylene dichloride)
4655	1,2-Dichloropropane
5215	1,3,5-Trimethylbenzene
9318	1,3-Butadiene
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
4735	1,4-Dioxane (1,4- Diethyleneoxide)
4836	1-Propene
5220	2,2,4-Trimethylpentane
4410	2-Butanone (Methyl ethyl ketone, MEK)
4538	2-Ethyltoluene
4860	2-Hexanone
4531	3-Ethyltoluene
4542	4-Ethyltoluene
4910	4-Isopropyltoluene (p-Cymene)
4995	4-Methyl-2-pentanone (MIBK)
4315	Acetone
4320	Acetonitrile
4325	Acrolein (Propenal)
4340	Acrylonitrile
4355	Allyl chloride (3-Chloropropene)
4357	alpha-Methylstyrene
6698	alpha-Pinene
4375	Benzene
5635	Benzyl chloride
4395	Bromodichloromethane
4400	Bromoform
4450	Carbon disulfide
4455	Carbon tetrachloride
4475	Chlorobenzene
4575	Chlorodibromomethane
4485	Chloroethane (Ethyl chloride)
4505	Chloroform
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4555	Cyclohexane
4560	Cyclohexanone
4625	Dichlorodifluoromethane (Freon-12)
9375	Di-isopropylether (DIPE)
6208	d-Limonene
4750	Ethanol

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Analyte Code	*Analyte*
4755	Ethyl acetate
4765	Ethylbenzene
4770	Ethyl-t-butylether (ETBE) (2-Ethoxy-2-methylpropane)
4835	Hexachlorobutadiene
4890	Isopropyl acetate
4895	Isopropyl alcohol (2-Propanol, Isopropanol)
4900	Isopropylbenzene
5240	m+p-xylene
4950	Methyl bromide (Bromomethane)
4960	Methyl chloride (Chloromethane)
4990	Methyl methacrylate
5000	Methyl tert-butyl ether (MTBE)
4975	Methylene chloride (Dichloromethane)
5005	Naphthalene
4425	n-Butyl alcohol (1-Butanol, n-Butanol)
4415	n-Butyl-acetate
4435	n-Butylbenzene
5875	n-Decane
6235	n-Dodecane
4825	n-Heptane
4855	n-Hexane
5026	n-Nonane
5027	n-Octane
5090	n-Propylbenzene
6747	n-Undecane
5250	o-Xylene
4440	sec-Butylbenzene
5100	Styrene
4370	T-amylmethylether (TAME)
4420	tert-Butyl alcohol
4445	tert-Butylbenzene
5115	Tetrachloroethylene (Perchloroethylene)
5120	Tetrahydrofuran (THF)
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
5225	Vinyl acetate
5235	Vinyl chloride
5260	Xylene (total)

EPA TO-17 **10312206** **Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes**

Analyte Code	*Analyte*
5160	1,1,1-Trichloroethane
5110	1,1,2,2-Tetrachloroethane
5195	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
5165	1,1,2-Trichloroethane
4630	1,1-Dichloroethane
4640	1,1-Dichloroethylene
5155	1,2,4-Trichlorobenzene
5210	1,2,4-Trimethylbenzene
4570	1,2-Dibromo-3-chloropropane (DBCP)
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
4695	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane (Ethylene dichloride)
4655	1,2-Dichloropropane
5215	1,3,5-Trimethylbenzene
9318	1,3-Butadiene

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Analyte Code	*Analyte*
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
4735	1,4-Dioxane (1,4- Diethyleneoxide)
5220	2,2,4-Trimethylpentane
4410	2-Butanone (Methyl ethyl ketone, MEK)
4860	2-Hexanone (MBK)
4995	4-Methyl-2-pentanone (MIBK)
4315	Acetone
4320	Acetonitrile
4375	Benzene
4395	Bromodichloromethane
4400	Bromoform
4450	Carbon disulfide
4455	Carbon tetrachloride
4475	Chlorobenzene
4575	Chlorodibromomethane
4485	Chloroethane (Ethyl chloride)
4505	Chloroform
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4555	Cyclohexane
4625	Dichlorodifluoromethane (Freon-12)
4750	Ethanol
4765	Ethylbenzene
4835	Hexachlorobutadiene
4895	Isopropyl alcohol (2-Propanol, Isopropanol)
4900	Isopropylbenzene
5240	m+p-xylene
4960	Methyl chloride (Chloromethane)
5000	Methyl tert-butyl ether (MTBE)
4975	Methylene chloride (Dichloromethane)
5005	Naphthalene
4825	n-Heptane
4855	n-Hexane
5027	n-Octane
5250	o-Xylene
5100	Styrene
5115	Tetrachloroethylene (Perchloroethylene)
5120	Tetrahydrofuran (THF)
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
5235	Vinyl chloride
5260	Xylene (total)

EPA TO-4A **10249204** **Pesticides and PCBs by HV PUF GC**

Analyte Code	*Analyte*
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
8880	Aroclor-1016 (PCB-1016)
8910	Aroclor-1260 (PCB-1260)
7115	beta-BHC (beta-Hexachlorocyclohexane)
7105	delta-BHC
7470	Dieldrin

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Analyte Code	Analyte
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide
7810	Methoxychlor

MADEP APH **90017188** **Air-Phase Petroleum Hydrocarbons**

Analyte Code	Analyte
9318	1,3-Butadiene
3792	APH Aliphatics C5-C8
3793	APH Aliphatics C9-C12
3794	APH Aromatics C9-C10
4375	Benzene
4765	Ethylbenzene
5240	m+p-xylene
5000	Methyl tert-butyl ether (MTBE)
5005	Naphthalene
5250	o-Xylene
5140	Toluene

ALS Standard Operating Procedure

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DOCUMENT TITLE:	ANALYSIS OF HALOGENATED VOLATILE ORGANIC COMPOUNDS IN EMISSIONS FROM STATIONARY SOURCES USING GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTION (ECD) IN ACCORDANCE WITH A MODIFICATION OF CARB METHOD 422
REFERENCED METHOD:	CARB 422 MODIFIED
SOP ID:	SVO-CARB422
REV. NUMBER:	05.0
EFFECTIVE DATE:	04/25/2015

STANDARD OPERATING PROCEDURE

ANALYSIS OF HALOGENATED VOLATILE ORGANIC COMPOUNDS IN EMISSIONS FROM STATIONARY SOURCES USING GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTION (ECD) IN ACCORDANCE WITH A MODIFICATION OF CARB METHOD 422

CARB 422 MODIFIED

SOP ID:	SVO-CARB422	Rev. Number:	05.0	Effective Date:	04/25/2015

Approved By: _____ Date: 4/14/15
SVOC Team Leader – Madeleine Dangazyan

Approved By: _____ Date: 4/20/15
QA Manager – Chaney Humphrey

Approved By: _____ Date: 4/15/15
Laboratory Director – Kelly Horiuchi

Archival Date:	_____	Doc Control ID#:	Non-Controlled	Editor:	_____

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ANALYSIS OF HALOGENATED VOLATILE ORGANIC COMPOUNDS IN EMISSIONS FROM STATIONARY SOURCES USING GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTION (ECD) IN ACCORDANCE WITH A MODIFICATION OF CARB METHOD 422

1) Scope and Applicability

1.1 This gas chromatograph method is used in the analysis of chloroform, trichloroethene, and tetrachloroethene by a modification of CARB Method 422. Other compounds that maybe reported provided that the requirements of this document are followed are: carbon tetrachloride, 1,2-dichloroethane, 1,2-dibromoethane, trichlorofluoromethane, 1,3-butadiene and dichloromethane. This method cannot be used to determine compounds of high molecular weight, compounds that may polymerize before analysis or compounds that have very low vapor pressures at stack or instrument conditions.

1.2 This method applies to but is not limited to the following sample matrices: ambient air, source emissions, landfill gases, digester gases, and vehicular exhaust. The range of this method for quantifying target analyte gases, depending on the concentration of the samples, is approximately 0.0010 to 200ppm. The upper limit may be extended by diluting the sample with an inert gas or by using a smaller injection volume. Approximately twenty samples may be analyzed in one eight hour day.

2) Summary of Procedure

2.1 Samples are collected in Tedlar bags, and delivered to the laboratory for analysis. A modification of the method may be used for the collection of samples in Summa canisters or glass bottles. An aliquot is drawn from the sampling container using a gastight syringe and injected onto a chromatographic column where the analytes are separated and measured using an electron capture detector (ECD). Analytes are identified and quantified based on their retention time, which is compared with that of a known standard under identical conditions. The Tedlar bag sampling and analysis is not suitable for monitoring 1,3-butadiene in combustion source emissions. Refer to CARB Method 422.102 for the analysis of 1,3-butadiene.

3) Definitions

3.1 Relative Standard Deviation (RSD) The RSD is the coefficient of variation (CV; ratio of the standard deviation to the mean) multiplied by 100 to convert the CV to a percentage of the mean.

3.2 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.3 Field Sample A sample collected and delivered to the laboratory for analysis.

3.4 Batch QC Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD), etc.

3.5 Calibration Standard (Initial Calibration – ICAL) A calibration standard is a known concentration of desired analyte(s) prepared from a primary standard, which is, in turn, prepared from a stock standard material. A calibration standard is analyzed at varying concentrations and used to calibrate the response of the measurement system with respect to analyte concentration.

3.6 Initial Calibration Verification (ICV) Standard An initial calibration verification standard (ICV) is a standard that is prepared from materials obtained from a source other than

the source for the calibration standards and is analyzed after the measurement system is calibrated, but prior to sample analysis in order to verify the calibration of the measurement system.

3.7 Continuing Calibration Verification (CCV) Standard A continuing calibration verification standard (CCV) is a midrange calibration standard that is analyzed periodically to verify the continuing calibration of the measurement system.

3.8 Method Blank (MB) The method blank (MB) for this method is ultra-pure nitrogen that is analyzed to verify the zero point of the analytical system and to verify freedom from carryover.

3.9 Method Reporting Limit (MRL) The minimum reliably quantifiable concentration of a compound.

3.10 Laboratory Control Sample (LCS) For the purposes of this document, a laboratory control sample (LCS) shall be a calibration standard of known concentration. The percent recovery of the analyte(s) in the LCS is used to assess method performance.

3.11 Laboratory Duplicate Aliquots of a sample taken from the same container under laboratory conditions which are processed and analyzed independently.

3.12 Precision Precision of a method is how close results are to one another, and is usually expressed by measures such as standard deviation, which describe the spread of results.

3.13 Bias The bias of a method is an expression of how close the mean of a set of results (produced by the method) is to the true value.

3.14 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop “ticks” have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

4) Health and Safety Warnings

4.1 Pollution Prevention and Waste Management All waste management must be carried out in accordance with the most recent version of the *SOP for Waste Disposal*.

4.2 This procedure may include CHEMICAL, OPERATIONAL and/or EQUIPMENT hazards. Employees must review and understand the following hazards and their preventive measures prior to proceeding with this activity. Hazard information related to this activity which is not included or referenced in this document should be immediately brought to the attention of the Department Supervisor.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Standard and sample preparation.	Exposure to potential health hazards through absorption through skin. Inhalation hazards.	Reduce exposure through the use of gloves and fume hoods. Safety glasses must be worn when working in the prep lab. Care should be taken when handling standard material in a neat or highly concentrated form. Personal protective clothing (safety glasses, gloves, and lab coat) are required when handling standard material in neat form. Consult Safety Data Sheets (SDS) for compounds being handled in this procedure, and be familiar with proper safety precautions. SDS shall be reviewed as part of employee training. Refer to the laboratory's *Environmental Health and Safety Manual* for additional information regarding safety in the workplace.
Job Task #2:	**Hazards**	**Preventative Measures**
Using and moving compressed gas cylinders.	Gas leak, fire, and explosion. Personal injury due to falling during transport.	All cylinders must be secured in an upright position to a wall or immovable counter with a chain or a cylinder clamp when not in use. Keep safety caps on when cylinders are not in use. A handcart must be used when transporting cylinders. The cylinder must be secured to the handcart with a chain or belt. Full cylinders must be kept separate from empty cylinders. Flammable gases (i.e. pressurized hydrogen) must be clearly labeled. Flammables and oxidizers must be separated by a ½-hour fire wall or by at least twenty feet.
Job Task #3:	**Hazards**	**Preventative Measures**
Glass syringe use	Skin lacerations and punctures.	The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.
Job Task #4:	**Hazards**	**Preventative Measures**
Working with and Pressurization of glass bottles	Personal injury from breakage or shattering.	Wear safety glasses when working with glass bottles. Gloves may be worn to help maintain grip. Bottle Vacs must not be pressurized higher than 7 psig.

5) Cautions

5.1 An instrument maintenance log shall be kept documenting maintenance performed on each analytical system. This log must be kept current. The serial numbers of each instrument shall be recorded in the front of the logbook. An entry shall be made in the appropriate log every time maintenance is performed. The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, or cleaning the source. The entry in the log must include:

(a) the date of maintenance
(b) who did the maintenance
(c) description of the maintenance
(d) proof that the maintenance activity was successful

A notation of a successful continuing calibration or initial calibration shall serve as proof that the maintenance is complete and the instrument is in working order.

5.2 Carrier Gas Purifier

If in-line purifiers or scrubbers are in place, these purifiers must be changed as recommended by the supplier.

5.3 GC System

5.3.1 Column Performance should be monitored by observing peak shapes and column bleed. Over time, the column may exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur depends on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Poor performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.3.2 Injection Port Injection port maintenance includes changing the injection port liner and column ferrule as needed. Liners should be changed when recent sample analyses predict a problem in chromatographic performance.

5.3.3 Injector Septa Septa should be changed monthly or whenever there is a noticeable change in peak definition. For best results with air analyses, two septa are placed into the injector in order to eliminate loss during manual injections.

5.3.4 Electron Capture Detector The ECD contains Nickel-63 and must undergo a radioactive leak or “wipe” test every 6 months. The radioactive leak test records are to be maintained for a minimum of 3 years. If a leak test fails, the ECD must be immediately taken out of use and the following must occur:

- Send detector to an authorized repair or disposal facility.
- Prepare and submit a report within 30 days to the California department of radiological health including a complete description of the device (manufacturer, type, serial number) and a brief description of the event and the remedial action taken (*California Code of Regulations).*

Under no circumstances is the ECD unit to be opened, cleaned, repaired or modified by laboratory personnel, as this would be a direct violation of the General License requirement.

6) Interferences

6.1 Contaminated Sample

Care must be taken to prevent ambient air intrusion into the sample container during canister pressurization and laboratory analysis. When using adapters and fittings the dead volume must be evacuated and replaced with the sample gas prior to sampling from the container. The sampling syringe shall then be flushed with the sample gas to remove residual ambient air. An aliquot greater than is needed is drawn, and the syringe plunger is adjusted to the appropriate volume *immediately* before injecting.

6.2 Carrier Gas Contamination

To prevent system contamination, UHP/ZERO grade helium (99.999% purity) is used as the carrier gas. Additionally, a purifier is incorporated into the analytical system as another precaution in preventing contamination.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review and reporting. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP must perform analysis and interpretation of the results. This demonstration shall be in accordance with the training program of the laboratory. The department supervisor/manager or designee shall perform final review and sign-off on the data.

7.2 Training shall be conducted in accordance with the *SOP for Training Policy.* An initial demonstration of proficiency shall be performed prior to independent analyses of samples. In addition, a continuing demonstration must be performed annually or prior to the analysis of samples. Both demonstrations consist of spiking Tedlar bags with the LCS standard and evaluating for both precision and accuracy. The criteria for approval are the same as the acceptance criteria for the LCS as specified in this document. See Attachment 1.

8) Sample Collection, Handling, and Preservation

8.1 The samples are collected and delivered to the laboratory for analysis in either Tedlar bags or specially prepared canisters, or glass sampling bottles (Bottle Vac. Entech Instruments). Samples collected in bags must be analyzed within 72 hours after sample collection unless otherwise specified by the client. Samples delivered in cleaned, evacuated summa or other specially prepared canisters do not have specified holding times for atmospheric gases but should be analyzed within 30 days from the date of collection.

9) Equipment and Supplies

9.1 Gas Chromatograph

HP 5890 or equivalent equipped with an electron capture detector, and having a temperature programmable oven. The column shall be 60m, 0.53mm ID RT_x-1 or equivalent with a 5µm film thickness.

Conditioning of the chromatographic column is required prior to use of the system. The column should be conditioned with a continuous flow of chromatographic grade helium and temperature programmed from 35°C to 200°C at a rate of five degrees per minute. The column should be held at 200°C for at least four hours.

9.2 Regulators

Regulators are used on the gas cylinders supplying the GC and for preparing cylinder standards.

9.3 Data System

A data system with the ability to collect data from the GC detector, integrate the peaks and perform the appropriate quantification calculations shall be used. This laboratory currently uses HP Chemstation/Enviroquant GC software.

9.4 Syringes

Gas tight syringes of the following volumes: 10mL, 1.0mL, and 0.5mL.

9.5 Tedlar Bags

New Tedlar bags are used for preparing standards and diluting very concentrated samples, which fall outside of the initial calibration range.

10) Standards and Reagents

10.1 All samples and standards must be stored separately. The concentration, preparation and expiration date as well as analyst's initials must be identified on the standard label. Each standard must also be uniquely identified with a laboratory ID number.

All certificates shall be noted with the standard identification number, date received and initials of the receiving analyst and retained by the quality assurance department.

10.2 Carrier and Calibration Standard Balance Gas

10.2.1 Helium - UHP/ZERO (99.999%) or higher in purity

10.2.2 Nitrogen - UHP/ZERO (99.999%) or higher in purity

10.3 Neat Standards

These standards must be stored in accordance with the requirements described in the *SOP for Handling Consumable Materials*. These standards may be stored for a period of 5 years for neat standards, 2 years for air standards or as recommended by the manufacturer.

Compound	Purity	MW	Density
Chloroform	99+%	119.4	1.4832
Trichloroethene	99+%	131.4	1.4642
Tetrachloroethene	99+%	165.8	1.6227
Carbon tetrachloride	99+%	153.8	1.5940
1,2-Dichloroethane	99+%	98.96	1.2351
1,2-Dibromoethane	99+%	187.9	2.17
Trichlorofluoromethane	99+%	137.4	1.494
1,3-Butadiene	99+%	54.09	0.6149
Dichloromethane (Methylene Chloride)	99+%	84.94	1.3266

10.4 Initial Calibration Standard / Working Standard

Prepare a neat cocktail standard from the above stated compounds by adding the appropriate volume of the neat compounds into a clean 2mL vial.

The current cocktail concentration recommendation is 200ppm for tetrachloroethene and 1000ppm for all other compounds. Determine the spike volume and add this amount to 1L of high purity nitrogen in a Tedlar bag. Record the calibration standard in accordance with the requirements described in the *SOP for Handling Consumable Materials.* Depending on the desired dynamic range of the initial calibration, various dilutions shall be made from this standard bag. A serial dilution may also be prepared from this standard bag.

The intermediate standard, along with all dilutions, must be stored at room temperature and expires 3 days after preparation.

10.4.1 *Equi-mass "soup"* (contains compounds in equal mass amounts) or *cocktail* prepared from neat compounds.

Cocktail Preparation:

Step 1: This cocktail is prepared by combining a calculated amount of each neat compound into a small glass vial based on the desired Tedlar bag standard concentration of 200ppm for tetrachloroethene and 1000ppm for other compounds. Use a microliter syringe to transfer each compound, cleaning with solvents in between. Put the vial in the freezer between aliquots to minimize volatilization. Take the density and molecular weight of each compound into account to determine the actual amount of each compound to spike into the cocktail by using the following equation.

$$S = \frac{\frac{S_A * MW * 1L * \frac{m^3}{1000L}}{24.46}}{D} \qquad \text{(Equation 1)}$$

Where:

S Calculated volume, per compound (uL)
MW Molecular weight for each compound, g/mole
S_A Desired concentration for each compound (ppm= mg/m³)
D Density (g/mL); refer to the density references

Example: The actual volume of chloroform to add to the cocktail is calculated by the following.

$$\text{S(Chloroform)} = \frac{\dfrac{1000mg/m^3 * 119.4 * 1L * \dfrac{m^3}{1000L}}{24.46}}{1.4832} = 3.29\text{uL}$$

Hint: To obtain a larger cocktail volume, multiply each compound volume by a multiplier (i.e., 40 or 50). This procedure prevents from having to prepare cocktail more often.

Tabulate all of the calculated spike amounts and spike the total into a 1L Tedlar bag filled with nitrogen. Place in an oven at approximately 60ºC for about 10 minutes. Allow the Tedlar bag to sit for about 15-20 minutes for an equilibrium period.

11) Method Calibration

11.1 Initial Calibration

The instrument must be calibrated initially and whenever the laboratory takes corrective action (maintenance), which may change or affect the initial calibration criteria, or if the continuing calibration acceptance criteria have not been met. Introduce each initial calibration concentration standard (at least five levels, analyzed from low concentration to high concentration) by direct injection using a gas tight syringe. Perform all calibration runs according to the analytical portion of the sample analysis described in Section 12.1

Note: The concentrations of the initial calibration may change as long as the low standard analyzed is the same as the reporting limit for each analyte.

11.1.1 Initial Calibration Requirements

Once a set of ICAL standards is analyzed, the previous ICAL may no longer be used to analyze new samples and it must be archived. The only time an archived ICAL can be used thereafter is to review or re-evaluate samples(s) previously processed using that ICAL.

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. Highest concentration, together with the lowest concentration, defines the calibration curve.
3. Lowest concentration must be at method reporting limit.
4. A blank should be analyzed prior to beginning the analysis of the calibration standards.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from low to high concentration.
8. All ICAL analyses must be completed within 48 hours.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.

- Lowest concentration must be at the MRL and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
- Points at high end may be dropped, but doing so lowers the calibration curve range.
- Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution or standard preparation error, or instrument malfunction) is accounted for and documented in a nonconformity and corrective action report (NCAR). In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action.
- If a point or a calibration standard is dropped, the reason must be documented (and the results maintained with the documentation for the final ICAL).
- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 48 hours).
- Once the ICAL has been used to calculate and report sample results, it is not to be changed.

11.1.2 Initial Calibration Review

Analyst's calculations and assessment along with a peer review of all ICAL data and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples. Sample results may only be reported if the ICAL is reviewed and found to be acceptable.

11.1.3 Initial Calibration File

An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- Blank analysis quantitation report
- Calibration status report (a.k.a. Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and evaluate continuing calibration report (a.k.a. Percent Difference Report)

11.1.4 Initial Calibration Verification

Verify the initial calibration by analyzing an independent calibration verification standard (ICV).

12) Sample Preparation/Analysis

12.1 Analytical Sequence and Data System Setup

12.1.1 Data System Load the appropriate acquisition method file for the gas chromatograph temperature program. Load the appropriate analytical sequence. Enter the analytical sequence information in the table window, including standard name, sample name and injection volume. Run the sequence and inject the standards and samples per the guidelines in Section 12.1.2.

12.1.2 Analytical Sequence The analytical batch must be completed for the analysis of ≤20 field samples. Laboratory duplicates (LD), duplicate field samples and sample dilutions are considered samples. Batch QC samples may be analyzed anywhere in the analytical sequence, with the exception of the method blank which must be analyzed prior to sample analysis in order to demonstrate a contamination free system.

Analytical Sequence Guideline[1]

Sample Description(w/ICAL)	*Sample Description*
Calibration Stds.[2]	CCV[3]
ICV[4]	MB[5]
MB[5]	LCS[6]
LCS[6]	Samples 1-10
Samples 1-10	CCV[3]
CCV[3]	Samples 11-19
Samples 11-19	LD[7]
LD[7]	CCV[3]
CCV[3]	

[1] The batch QC may be analyzed in an order other than the one listed in this document; the analytical sequence specified below is a guideline.
[2] The initial calibration must be generated in accordance with the guidelines detailed in Section 11.1 of this document.
[3] In cases, where the ICAL is not performed the analytical sequence must begin with the analysis of a CCV standard. In addition, the analytical sequence shall end with an acceptable CCV.
[4] Every ICAL must be followed by a second source standard (ICV) which contains all of the target analytes.
[5] The method blank must be analyzed prior to any samples within the sequence.
[6] Every analytical sequence must include a laboratory control sample. A LCS shall be analyzed at a rate of one per twenty samples or fewer for each analyte.
[7] A laboratory duplicate must be analyzed at a frequency of 1 in 20 or fewer samples.

12.2 GC Configuration

12.2.1 Temperature Program The GC oven temperature programming must be set to completely elute all of the target analytes. The temperature program ramps up to a high temperature, not exceeding the maximum temperature rating of the column in use, and holds there to allow all heavier compounds to elute, in order to prevent carryover to the next injection. The settings and system parameters are as follows.

Instrument Control Parameters – GC21	
Sample Inlet	GC
Injection Source	Manual
Run Time	12 minutes
Injector	
Mode	Packed
Temperature	150˚C
Pressure	18psi at 100˚C oven temperature
Isothermal Oven	
Initial Temperature	100˚C
Initial Time	12 minutes
Column	
Model Number	RTx-1
Nominal Length	60m
Nominal Diameter	0.53mm ID
Film Thickness	5µm
ECD	
N2	50mL/min
Temperature	280˚C

12.3 Continuing Calibration

A continuing calibration check shall be performed at the beginning, after every 10 samples and at the end of an analytical sequence, or every twenty field samples, not to exceed a 24-hour period. The concentration of the calibration verification may be varied within the established calibration range.

12.4 Method Blank

The method blank shall be obtained using ultra high purity nitrogen directly injected in the same manner as the standards and samples. A method blank must be analyzed prior to analysis of samples. A method blank must also be analyzed if carryover contamination is suspected.

12.5 Laboratory Control Sample

The laboratory control sample shall be an injection of the continuing calibration or initial calibration verification standard. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

12.6 Analysis

12.6.1 Canister and Glass Bottle Pressurization Sample analysis must be made using the same instrument parameters as that of the calibration standards. Refer to the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters* for the procedure of how canisters and glass bottles are to be pressurized prior to analysis. The analyst shall record the appropriate pressures on the Service Request form. This includes noting any anomalies for which the appropriate corrective actions have been detailed and must be followed accordingly.

12.6.2 Sample Analysis Sample analysis shall be performed by a direct injection technique using gas tight syringes. Insert the syringe through the Tedlar bag septum or summa can fit with an adapter. When using adapters and fittings the dead volume must be evacuated and replaced with the sample gas prior to

sampling from the container. The sampling syringe shall then be flushed with the sample gas to remove residual ambient air and vented into a waste bag. This procedure entails drawing an aliquot greater than is needed, and adjusting the syringe plunger to the appropriate volume *immediately* before injecting.

Note: The maximum allowed injection volume is 500uL

Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments). Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fittings higher than 80ºC.

12.6.3 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot or a dilution in a Tedlar bag must be analyzed. Guidance in performing dilutions and exceptions to this requirement are given below.

- Use results of the original analysis to determine the approximate dilution factor required getting the largest analyte peak within the initial calibration range.
- The dilution factor chosen should keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument. Additional compounds may be reported as long as they are within the calibration range.
- Analysis involving dilution should be made with high purity nitrogen and must be reported with a dilution factor.

Tedlar bag dilution:

- Calculate the sample amount and volume of balance gas needed to obtain the required dilution.
- Fill a new 1.0L Tedlar bag with nitrogen using the appropriate gas tight syringe.
- Remove the difference in the balance gas using the appropriate gas tight syringe.
- Add the calculated sample amount using a gas tight syringe.

12.7 Laboratory Duplicate

Analyze two separate aliquots from the same sample container. A laboratory duplicate must be analyzed a frequency of 1 in 20 field samples. The laboratory duplicate should be rotated among clients, whenever possible

12.8 Manual Integration

The integration(s) for each sample is checked to ensure that it has been integrated properly. Assuming an incorrect automatic integration the analyst shall conduct the manual integration in accordance with the *SOP for Manual Integration Policy* including all documentation and reviews associated with the process. The review should include the analyst and peer reviewer initialing and dating the manual integration as an indication of acceptability and approval.

12.9 Method Detection and Quantitation Limits

The MDL must be performed in accordance with the procedure outlined in the *SOP for the Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* Method detection limits must be determined annually and whenever

there is a change in the test method that affects how the test is performed, or when a change in instrumentation is such that it affects the sensitivity of the analysis. The MDL study shall be performed on each instrument for which this method is performed. All supporting data must be approved and retained.

12.10 Cleaning Tedlar Bags

Fill with nitrogen and evacuate several times. In the final cleaning step partially fill the bags with nitrogen and evacuate using a pump.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. Files should be named with a character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files shall be saved in a unique sub-directory on the server. An example of how the analyst should store analytical data is as follows:

Instrument Number/Data/Method ID/yr_month/.d*

The initial calibration curve may be saved with an identification such as CARB followed by the date of the analysis (mm,yy). This file should be saved in the following directory: J:\"instrument ID"\Method\. No curve may be overwritten at any time to ensure a complete audit trail.

14.2 Sufficient raw data records must be retained of the analysis, instrument calibrations and method detection limit studies including: analysis/calibration date and time, test method, instrument, sample identification, each analyte name, analyst's initials, concentration and response, and standards used for the analysis and calibrations, any manual calculations including sample dilutions and manual integrations. Information entered and reported on the quantitation report and instrument run log must be complete and accurate.

14.3 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date of analysis, time of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, any manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings), standard and reagent origin, sample receipt, calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, and assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 Initial Calibration

- Tabulate the linear relationship using Equation 1

15.2 Initial Calibration Verification

- Calculate the concentration for each analyte using equation number 1.
- Calculate the percent difference (%D) between the calculated concentration (equation number 1) and the actual concentration using equation number 2.

15.3 Continuing Calibration Verification

- Calculate the concentration of each analyte using equation number 1.
- Calculate the percent difference (%D) between the calculated concentration (equation number 1) and the actual concentration using equation number 2.

15.4 Laboratory Control Sample

- Calculate the concentration of each analyte using equation number 1.
- Calculate the percent recovery (%R) for each analyte using equation number 4.

15.5 Sample Analysis

- Calculate the concentration of each analyte using equation number 1.
- Calculate the dilution factor if necessary using equation number 5.

15.6 Laboratory Duplicate

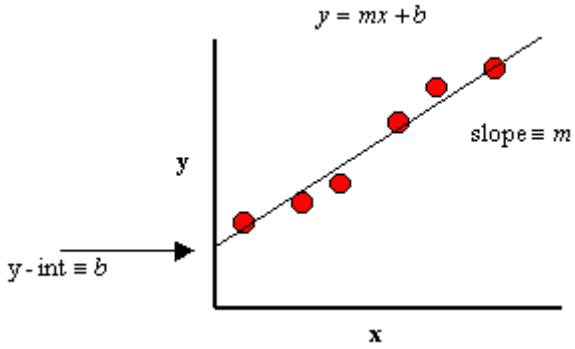
- Calculate the concentration of each analyte using equation number 1.
- Calculate the relative percent difference (RPD) using equation number 3.

15.7 Calculations

15.7.1 Equation Number 1

Linear Relationship

Say we have a set of data, (x_i, y_i), shown at the left. If we have reason to believe that there exists a **linear relationship** between the variables **x** and **y**, we can plot the data and draw a "best-fit" *straight line* through the data. Of course, this relationship is governed by the familiar equation $y = mx + b$. We can then find the **slope, *m*,** and **y-intercept, *b*,** for the data, which are shown in the figure below.



Linear Regression Equations

If we expect a set of data to have a linear correlation, **it is not necessary for us to plot the data** in order to determine the constants *m* (slope) and *b* (y-

intercept) of the equation $y = mx + b$. Instead, we can apply a statistical treatment known as **linear regression** to the data and determine these constants.

Given a set of data (x_i, y_i) with *n* data points, the slope, y-intercept and correlation coefficient, *r*, can be determined using the following: (Note that the limits of the summation, which are *i* to *n*, and the summation indices on *x* and *y* have been omitted.)

$$m = \frac{n\sum(xy) - \sum x \sum y}{n\sum(x^2) - (\sum x)^2}$$

$$b = \frac{\sum y - m\sum x}{n}$$

$$r = \frac{n\sum(xy) - \sum x \sum y}{\sqrt{\left[n\sum(x^2) - (\sum x)^2\right]\left[n\sum(y^2) - (\sum y)^2\right]}}$$

Casio fx-300W Calculation Instruction for Linear Regression:

The regression formula for linear regression is: y = A+Bx
Enter REG Mode (Linear Regression)
Hit Mode, 3 Reg, 1 Lin shift Scl = (memory clear)
Enter data points - concentration vs absorbance: [ex: 0,0 'M+', 0.1, 0.099 'M+', 0.2, 0.198 'M+' & 0.4, 0.402 'M+']
For correlation coefficient – hit 'Shift' then 'r' then '=' 0.999965116
For unknown concentrations enter absorbance (ex: 0.175 from spectrophotometer) - hit shift '+' key = 0.17524865

15.7.2 Equation Number 2

Percent Difference, %D,

The %D is used for evaluating ICV and CCV vs. the initial calibration

$$\%D = \frac{C_{CCVorICV} - C_{std}}{C_{std}}(100)$$

where, for any given analyte:

$C_{CCVorICV}$	is the concentration being evaluated
C_{std}	is the concentration from the current calibration curve

15.7.3 Equation Number 3

Relative Percent Difference (RPD)

$$\frac{|R_1 - R_2|}{\left(\frac{R_1 + R_2}{2}\right)} x100$$

where:

R_1 First measurement value
R_2 Second measurement value

15.7.4 Equation Number 4

Percent Recovery (%R):

$$\%R = \frac{C}{S} x100$$

Where:

C = Concentration of the analyte recovered
S = Spiked amount

15.7.5 Equation Number 5

Dilution Factor

$$DF = \frac{V_T}{V_S}$$

Where:

DF = dilution factor
V_S = volume of sample (mL) used
V_T = total volume of dilution (mL)

15.7.6 Equation Number 6

Results

In order to obtain the final reported value, the result must be adjusted with the canister dilution factor, any sample dilution and injection volume and converted to ug/m^3.

Example:

- R = Result = 22.079ppb (on column)
- DF = Dilution Factor = 1.58 (canister dilution factor)
- IV_N = Normal Injection Volume = 0.5mL (see method blank injection volume)
- IV_A = Actual Injection Volume = 0.1mL
- AD = Additional Dilution = 1000

$$ppbV = \frac{R * DF * IV_N * AD}{IV_A} = \frac{22.079 * 1.58 * 0.5 * 1000}{0.1} = \frac{17442}{0.1} = 174424 =$$

170,000ppbV

- MW is the molecular weight of PCE
- 24.46 is the molar volume of gas at lab conditions (constant)
- All results are reported with two significant figures

$$ug/m^3 = \frac{ppbV * MW}{24.46} = \frac{174424 * 165.8}{24.46} = 1{,}182{,}318 = 1{,}200{,}000 ug/m^3$$

15.8 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated following the data review checklist in Attachment 3. The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The data review checklist shall be used to document the review process. Once it has been completed, the checklist must be initialed, dated and filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.3.

15.9 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results. The analyst shall ensure that all of the requirements specified in this document and the *SOP for Data Review and Reporting* are followed.

15.10 Sample Preparation and Analysis Observations / Case Narrative Summary Form

The case narrative summary form, which is included in the *SOP for Laboratory Storage, Analysis, and Tracking*, must be generated when there are any specific sample composition information, sample preparation, analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved.

This form is necessary for documentation purposes and shall be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through preparation, analysis, data reduction, and reporting.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 This section contains technical acceptance criteria. To the extent possible, samples shall be reported only if all of the quality control measures are acceptable.

16.2 It must be determined if there are any instrumentation problems contributing to the occurrence of any out-of-control data. If it is decided that problems do exist, then the analyst must determine if the effects have caused any modification in the data from

client submitted samples. This being the case, all samples (including QC) that are affected by instrumentation problems must be re-analyzed following any necessary maintenance activity. All corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate.

16.3 Initial Calibration

16.3.1 Acceptance Criteria

- The correlation coefficient must be at least 0.98 from the least squares fit for the calibration to be considered acceptable.
- The retention time of each analyte at each calibration level must be within 0.1 minute of the midpoint standard in the calibration curve.

16.3.2 Corrective Action Inspect the system for possible sources. It may be necessary to change the column or take other corrective actions. Also, check standards for a bad injection and re-analyze standard. If a bad injection is not evident, perform maintenance and attempt another initial calibration (make notation in maintenance logbook regarding any steps taken). A demonstration of an in-control system is required before proceeding with the analysis.

Note: No ICAL may be interrupted by any maintenance procedure. Therefore, all standards incorporated in a curve must be reanalyzed.

16.4 Initial Calibration Verification Standard (ICV)

16.4.1 Acceptance Criteria

- The percent difference (%D) for each calculated target analyte must be within ±30% of the actual concentration of the standard.
- The retention time of each target analyte must be within 0.1 minute of the midpoint standard in the calibration curve.

16.4.2 Corrective Action The initial calibration verification should be re-analyzed. A second failed ICV must initiate corrective action and two consecutive ICVs must pass in order for the ICAL to be deemed acceptable. It may be necessary to prepare either new ICAL or ICV standards or both, perform maintenance and reanalyze the initial calibration.

16.5 Continuing Calibration Verification (CCV)

16.5.1 Acceptance Criteria

- The percent difference (%D) for each calculated target analyte must be within ±30% of the actual concentration.

16.5.2 Corrective Action If the criteria are not met, reanalyze (no more than two injections may be made before corrective action is initiated) or prepare a fresh CCV standard and reanalyze. If routine corrective action procedures fail to produce an acceptable calibration verification, a new initial calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data only under the following special condition:

When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the sample affected by the unacceptable CCV shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

16.6 Method Blank

16.6.1 Acceptance Criteria

- The method blank result for any target analyte must not be greater than the method reporting limit or contribute more than 10% of the sample concentration.

16.6.2 Corrective Action The source of the problem must be investigated and measures taken to eliminate the cause. Determine whether the contamination is from the instrument or due to contamination in the nitrogen, syringe or other source. Regardless, appropriate corrective measures must be taken and documented before further sample analysis proceeds. If the results are the same, the blank along with all associated samples must be reported to the client with the appropriate qualifier.

16.7 Laboratory Control Sample (LCS)

16.7.1 Acceptance Criteria

- The percent recovery for all compounds must be within 70% and 130%.

16.7.2 Corrective Action Determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and reanalyze the associated sample(s). If the problem is with the injection, reanalyze the LCS. If the results are still unacceptable and there does not appear to be any instrumentation problems refer to the appropriate reporting information.

16.8 Sample Analysis

16.8.1 Acceptance Criteria

- Sample results must be quantitated from the current instrument initial calibration and may not be quantitated from any continuing calibration verification standard.
- The field samples must be analyzed along with a laboratory method blank that has met the method blank criteria.
- All target analyte peaks must be within the initial calibration range.
- The retention time of each target analyte must be within 0.1 minute of the CCV.

16.8.2 Corrective Action To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out-of-control, and the data must be reported, all samples associated with the out-of-control quality control measures shall be reported with the appropriate data qualifier(s).

- When corrective actions are made, samples analyzed while the system was not functioning properly must be reanalyzed.
- Results not bracketed by initial instrument calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags.

16.9 Laboratory Duplicate

16.9.1 Acceptance Criteria

- The selected samples must be rotated among client samples so that various matrix problems may be noted and/or addressed.

- The results must meet all of the criteria for sample analysis as well as be <15% relative percent difference for all analytes of interest, provided that the concentration is greater than 10x the RL.

16.9.2 Corrective Action The sample(s) should be re-analyzed whenever the duplicate results are outside the technical acceptance window. If the results are still unacceptable and there does not appear to be any matrix effects, interfering peaks, or instrument problems, the results for both injections shall be reported to the client with the appropriate qualifier.

16.10 Samples Holding Time Expired The client is to be notified (best attempt) that the sample's holding time was missed and the client is to decide if the sample analysis shall continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 If a quality control measure if found to be out-of-control and the data must be reported, all samples associated with the out-of-control quality control measure shall be reported with the appropriate data qualifier(s).

18.2 Analysis quality control results (CCV, MB, LD, and LCS recoveries) out-of-control

If the associated samples are within holding time, re-analyze the sample. Alternatively, evaluate the effect on the sample results and report the results with qualifiers and/or discuss in the case narrative as detailed below.

18.2.1 CCV The LCS should be in control in order for any results to be reported with an out-of-control CCV (biased high). Refer to Section 16.5.

18.2.2 Method Blank If an analyte in the blank is found to be out-of-control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report. If the analyte is found in the blank but not in the sample and all other quality control meets acceptance criteria then the results for the sample may be reported without a qualifier. However, if other QC is out-of-control then an evaluation must be made and the results reported accordingly.

18.2.3 Laboratory Control Sample If the samples are analyzed with an out-of-control LCS, then all reported analytical results must be "flagged" with the appropriate data qualifier and/or discussed in the case narrative.

18.2.4 Laboratory Duplicate The appropriate data qualifier must be included for results associated with an out-of-control laboratory duplicate and/or discussed in the case narrative.

18.3 Sample quality control results out-of-control

Examine the sample results for matrix interference and for carryover. Reanalyze the sample(s) and/or reanalyze the sample(s) at a lower aliquot. If the out-of-control

results are due to matrix interference, report the results with a matrix interference qualifier.

Holding time qualifiers must be reported on samples not analyzed within holding time.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
05.0	04/25/15	C. Humphrey	Section 1.1 – Removed 1,1,1-trichloroethane
			Section 2.1 – Added glass bottles
			Section 4 – Revised
			Section 8.1 – Added glass bottles
			Section 10.3 – Removed 1,1,1-trichloroethane
			Section 12.6.1 – Revised; added glass bottles
			Table 1 – Removed 1,1,1-trichloroethane; updated MDL values

21) References and Related Documents

21.1 State of California Air Resources Board, Method 422 "Determination of Volatile Organic Compounds in Emissions from Stationary Sources", Amended December 13, 1991.

21.2 *SOP for Making Entries onto Analytical Records*, SOP ID CE-QA007

21.3 *SOP for Data Review and Reporting*, SOP ID ADM-DATA_REV

21.4 *SOP for Nonconformance and Corrective Action*, SOP ID CE-QA008

21.5 *SOP for Handling Consumable Materials,* SOP ID ADM-CONSUM

21.6 *SOP for Training Policy,* SOP ID CE-QA007

21.7 *SOP for Laboratory Storage, Analysis, and Tracking,* SOP ID ADM-LabSAT

21.8 *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation,* SOP ID CE-QA011

21.9 *SOP for Manual Integration Policy,* SOP ID CE-QA002

21.10 *SOP for Evaluation & Pressurization of specially Prepared Stainless Steel Canisters,* SOP ID SMO-Can_Press

22) Appendix

22.1 Tables

Table 1 – Target Analytes with Corresponding Method Detection and Reporting Limits

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Data Review Checklist

TABLE 1

CARB Method 422 (Modified) Target Analytes with Method Reporting Limits

Analyte	MDL (ppb)	MRL (ppb)
Chloroform	0.095	1.0
Trichloroethene	0.042	1.0
Tetrachloroethene	0.061	0.20
1,2-Dibromoethane	0.18	0.50

Note: These values may change with each new MDL study performed. Additional compounds must have a complete MDL study and the MRL must be at or higher than the low standard of the initial calibration.

Attachment 1

Training Plan

Training Plan for Analysis of Various Halogenated Compounds by GC/ECD

Trainee _____ Trainer _____ Completion Date _____ Instrument ____

1. Read SOP — Trainer ____ Trainee ____ Date _____
2. Read Method: CARB 422 — Trainer ____ Trainee ____ Date _____
3. Demonstrated understanding of the scientific basis of the analysis
   Gas chromatography Electron Capture Detector — Trainer ____ Trainee ____ Date _____
4. Demonstrated familiarity with related SOPs — Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences; Rev.___
   SOP for Making Entries onto Analytical Records; Rev.___
   SOP for Manual Integration Policy; Rev.___
   SOP for Significant Figures; Rev.___
   SOP for Nonconformance and Corrective Action; Rev.___
   SOP for Performing MDL Studies and Establishing Limits of Detection & Quantitation; Rev.___
5. Observe performance of SOP — Trainer ____ Trainee ____ Date _____
   ___standard preparation (gas-phase dilutions)
   ___sample preparation
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant introduction
   ___data reduction and reporting
6. Perform SOP with supervision — Trainer ____ Trainee ____ Date _____
   ___standard preparation (gas-phase dilutions)
   ___sample preparation
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant use
   ___data reduction and reporting
7. Independent performance of the SOP — Trainer ____ Trainee ____ Date _____
   ___standard preparation (gas-phase dilutions)
   ___sample preparation
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant proficiency
   ___data reduction and reporting
   ___initial demonstration of competency
   ___ four consecutive laboratory control samples
8. Instrument operation and maintenance — Trainer ____ Trainee ____ Date _____
   ___GC and capillary column installation
   ___ECD setup and maintenance
   ___data system

Attachment 2
Initial Calibration Checklist

Initial Calibration Checklist
Analysis of Various Halogenated Compounds by Modified CARB 422

ICAL Date: _____

Instrument: ☐ GC21 ☐ _____

Analyst			Reviewer
☐	1.	Is the required documentation in the ICAL file?	☐
		☐ Sequence report	☐
		☐ Blank analysis Quantitation Report	☐
		☐ Calibration Status Report (aka Calibration History) - Initial	☐
		☐ Coefficient of Determination	☐
		☐ Quantitation Report for each calibration standard (including manual integration documentation – before and after printouts)	☐
		☐ ICV Quant Report and Evaluate Continuing Calibration Report (aka Percent Diff. report)	☐
☐	2.	ICAL performed continuously (i.e., not interrupted for maintenance or sample analysis)?	☐
☐	3.	ICAL performed within 48 hours?	☐
☐	4.	Standards analyzed from low concentration to high concentration?	☐
☐	5.	All analytes in blank analysis <MRL?	☐
☐	6.	Does each analyte's ICAL include a minimum of 5 concentrations?	☐
☐	7.	For each analyte, is there only one value used for each calibration level?	☐
☐	8.	If a point is dropped, is information noted in the ICAL explaining the reason?	☐
☐	9.	Does this follow the point dropping policy (including re-analysis within 48 hrs)?	☐
☐	10.	For each analyte, is the lowest standard's concentration at or below the MRL?	☐
☐	11.	For each analyte, does the ICAL include 5 consecutive levels?	☐
☐	12.	For each analyte, are there no levels skipped?	☐
☐	13.	Does the calibration curve give a correlation coefficient ≥0.98?	☐
☐	14.	For the ICV analysis, is the percent recovery for each analyte 70-130%?	☐
☐	15.	Are all peak integrations including manual integrations (per *SOP for Manual Integration Policy*) acceptable? ***If so, initial and date the appropriate pages***	☐

COMMENTS:

_____

_____

_____

Reviewed By _____ Secondary Reviewer _____

Date _____ Date _____

Attachment 3

Data Review Checklist

Data Review Checklist
Modified CARB 422

(Note exceptions in Comments section and attach Sample Preparation and Analysis Observations / Case Narrative Summary as applicable)

Analysis Date_____ Client _____ QC Level _____

Project # _____ Due Date _____ Instrument ☐ GC21 ☐ _____

Analyst **Reviewer**

Initial Calibration

☐ 1. Is the referenced ICAL the most recent ICAL performed? ☐

☐ 2. Has the referenced ICAL been peer reviewed and all associated documentation including the ICAL review checklist available for review? ☐

☐ 3. Were all associated requirements within the specified limits? ☐

Continuing Calibration

☐ 4. CCV raw data submitted? ☐

☐ 5. Was the %D for the CCV ±30% (first or second injection)? ☐

☐ 6. CCV analyzed at the beginning of the sequence, every 10 samples, and the end of the sequence? ☐

Sample Data

☐ 7. Is all sample data present and correct? ☐

- ☐ Sample raw data?
- ☐ Target analyte responses within calibration range?
- ☐ Peak integrations acceptable?
- ☐ All manual integrations flagged and properly documented? If so, initial and date.
- ☐ Any essential retention time shifts?
- ☐ All calculations correct?
- ☐ First quantitation report initialed and dated by analyst?

QC Data

☐ 8. Duplicate sample analyzed 1 per 20 or fewer samples? ☐

☐ 9. Is the laboratory duplicate within 15% of their average? ☐

☐ 10. Is the LCS/LCSD within ±15% of their average (where applicable)? ☐

☐ 11. Is the recovery for the LCS and/or LCSD within 70-130%? ☐

☐ 12. Are all analytes in the MB < MRL? ☐

Reporting Information

☐ 13. Sample Preparation and Analysis Observations / Case Narrative Summary completed if applicable? ☐

☐ 14. Appropriate flags indicated on a Sample Preparation and Analysis Observations / Case Narrative Summary form when applicable? ☐

☐ 15. Reporting spreadsheet complete and all flags correctly indicated? ☐

COMMENTS: _____

_____

_____

Reviewed By _____ Secondary Reviewer _____

Date _____ Date _____

ALS Standard Operating Procedure

Proprietary - Uncontrolled Copy

DOCUMENT TITLE:	DETERMINATION OF HYDROGEN, CARBON MONOXIDE, CARBON DIOXIDE, NITROGEN, METHANE, AND OXYGEN USING GAS CHROMATOGRAPHY WITH THERMAL CONDUCTIVITY DETECTION (TCD) IN ACCORDANCE WITH EPA METHOD 3C OR ASTM D 1946
REFERENCED METHOD:	EPA METHOD 3C, ASTM D 1946
SOP ID:	VOA-EPA3C
REV. NUMBER:	13.0
EFFECTIVE DATE:	12/31/2015

STANDARD OPERATING PROCEDURE

DETERMINATION OF HYDROGEN, CARBON MONOXIDE, CARBON DIOXIDE, NITROGEN, METHANE, AND OXYGEN USING GAS CHROMATOGRAPHY WITH THERMAL CONDUCTIVITY DETECTION (TCD) IN ACCORDANCE WITH EPA METHOD 3C OR ASTM D 1946

EPA METHOD 3C, ASTM D 1946

SOP ID:	VOA-EPA3C	Rev. Number:	13.0	Effective Date:	12/31/2015

Approved By: _____ Date: 12/15/15
Department Supervisor - Wade Henton

Approved By: _____ Date: 12/15/15
QA Manager - Charley Humphrey

Approved By: _____ Date: 12/15/15
Laboratory Director - Kelly Horiuchi

Archival Date: _____ Doc Control ID#: Non-Controlled Editor: _____

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DETERMINATION OF HYDROGEN, CARBON MONOXIDE, CARBON DIOXIDE, NITROGEN, METHANE, AND OXYGEN USING GAS CHROMATOGRAPHY WITH THERMAL CONDUCTIVITY DETECTION (TCD) IN ACCORDANCE WITH EPA METHOD 3C OR ASTM D 1946

1) Scope and Applicability

1.1 The referenced method (EPA Method 3C) was written for the analysis of carbon dioxide, methane, nitrogen and oxygen, in municipal solid-waste landfill gas and other stationary sources but is easily modified for the gas chromatographic method determination of hydrogen and carbon monoxide. In contrast, the practice ASTM D 1946 covers the determination of the chemical composition of reformed gases and similar gaseous mixtures containing each of these six components. Method ASTM D 1945-03 modified which describes the analysis of natural gas may also be referenced.

1.2 This method is appropriate for quantifying target analyte gases depending on the concentration of the samples from approximately 500 ppmv to high percent values. The number of samples, which may be analyzed in one eight hour day, is approximately twenty. The reporting limits for these analytes are listed in Attachment 4 of this standard operating procedure.

2) Summary of Procedure

2.1 The EPA Method 3C was written for use with backfilled summa canisters but is easily modified for samples collected as vapor in Tedlar bags, steel tanks, glass bottles, summa or other specially prepared canisters. In contrast, the ASTM methods do not specify a requirement for the sampling container.

2.2 An aliquot is drawn from the sampling container using a sample loop and injected onto a packed chromatographic column where the analytes are separated and measured using a thermal conductivity detector (TCD). Samples are analyzed in duplicate for EPA Method 3C, but a modification may be made which entails a single injection per submitted field sample. However, results from samples analyzed per ASTM D 1946 are obtained using a single injection technique.

Note: Refer to Sections 12.13 and 15.9 for the list of reporting modifications for these methods.

3) Definitions

3.1 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.2 Field Sample A sample collected and delivered to the laboratory for analysis.

3.3 Batch QC The QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) or Laboratory Duplicate (LD).

3.4 Calibration Standard (Initial Calibration – ICAL) A calibration standard of a known concentration containing desired analyte(s) prepared from a primary standard, which is, in turn, prepared from a stock standard material. A calibration standard is injected at varying volumes and used to calibrate the response of the measurement system with respect to analyte concentration.

3.5 Initial Calibration Verification (ICV) Standard An ICV is a standard that is obtained from a source other than the source for the calibration standards and is analyzed after the

measurement system is calibrated, but prior to sample analysis in order to verify the initial calibration of the measurement system.

3.6 Method Blank (MB) An analyte-free matrix, which is carried through the entire analytical process. It is used to evaluate the process for contamination from the laboratory.

3.7 Laboratory Control Sample (LCS) An LCS is a standard that is obtained from a source other than the source for the continuing calibration verification standard (CCV). The percent recovery of the analyte(s) in the LCS is used to assess method performance.

3.8 External Standard Calibration External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas or peak heights are compared to peak areas or peak heights of the standards.

3.9 Analytical Batch A group of samples which behave similarly with respect to the sampling or the test procedures being employed and are processed as a unit using the sample lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. In an analytical batch of samples, the time period is 24 hours or up to twenty sample injections, whichever comes first of continuous operation without interruption.

3.10 Continuing Calibration Verification (CCV) Standard A continuing calibration verification standard is a midrange calibration standard that is analyzed periodically to verify the continuing calibration of the measurement system.

3.11 Precision Precision of a method is how close results are to one another, and is usually expressed by measures such as standard deviation, which describe the spread of results.

3.12 Bias The bias of a method is an expression of how close the mean of a set of results (produced by the method) is to the true value.

3.13 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop “ticks” have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.14 Ambient Air Ambient air within the laboratory which is sampled and analyzed once per batch to assess injector performance.

3.15 Limit of Detection (LOD) The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DoD Clarification). For consistency purposes, the LOD may be referred to as the MDL once it is reported; however, full verification will be on file in the laboratory per the procedures detailed in this document.

3.16 Limit of Quantitation (LOQ) The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. (DoD Clarification). For consistency purposes and since the LOQ and MRL are equivalent with regards to laboratory procedure, the LOQ will be referred to as the MRL in this document and once it is reported. Full verification will be on file in the laboratory per the procedures detailed in the document.

3.17 Detection Limit (DL) / Method Detection Limit (MDL) The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type 1 error) is 1%. (DoD Clarification). For consistency purposes, the DL may be referred to as MDL. Also, as far as reporting is concerned the MDL will be raised (where necessary) to the verified LOD per the procedures defined in this document and reported accordingly.

4) Health and Safety Warnings

4.1 Each compound, mixture of compounds, standards, as well as samples, should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest level possible through the use of hoods (to minimize inhalation). For proper handling, use, and disposal refer to the laboratory's *Environmental Health and Safety Manual*, Safety Data Sheets (located in the safety cubicle in the front office), as well as the *SOP for Waste Disposal*.

4.2 Safety Data Sheets (SDS) Safety Data Sheets (SDS) are available in the Safety cubicle located in the front office and shall be reviewed as part of employee training.

4.3 Safety Glasses Safety glasses are required when performing maintenance on pressurized systems.

4.4 Pressurized Gases The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. The regulator should not remain on size "D" cylinders when not in use. Sources of flammable gases (i.e. pressurized hydrogen) should be clearly labeled.

4.5 Pollution Prevention and Waste Management All waste management must be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal* as well as the *Environmental Health and Safety Manual*.

5) Cautions

5.1 A maintenance log shall be kept documenting maintenance performed on each analytical system and the instrument maintenance log must be kept current and reviewed quarterly. The serial numbers of each instrument shall be recorded in the front of the logbook. An entry must be made in the appropriate log each time any maintenance activity is performed (no matter the extent). The entry in the log must include:

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful continuing calibration or initial calibration shall serve as proof that the maintenance is complete and the instrument is in working order.

5.2 Carrier Gas Purifier If in-line purifiers or scrubbers are in place, these purifiers must be changed as recommended by the supplier.

5.3 GC System

5.3.1 Column Column performance should be monitored by observing peak shapes and column bleed. Over time, the column may exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur depends on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be changed or the packing replaced (see Section 9.1.1). Care should be taken to minimize the introduction of air or oxygen into the column whenever GC maintenance is performed.

Decreasing performance can also be due to a leak in the system. Leaks can be detected with the use of a leak detector. Fittings may need to be tightened or ineffective column ferrules replaced to eliminate any leak detected.

5.3.2 Detector Replace filament assembly as needed.

5.3.3 Injection Lines Purge with nitrogen to ensure the line is not blocked.

6) Interferences

6.1 Contamination Dry ambient air at sea level contains 78.08% Nitrogen, 20.95% Oxygen, 0.93% Argon, and approximately 0.033% Carbon Dioxide by volume. Precautions must be taken to prevent intrusion of ambient air into the analytical system and the sampling containers.

6.1.1 Contamination in the Sample Care must be taken to prevent ambient air intrusion into the sample container during canister pressurization and laboratory analysis. When using adapters and fittings the dead volume should be evacuated and replaced with the sample gas prior to sampling from the container.

6.1.2 Carrier Gas Contamination To prevent system contamination, UHP/ZERO grade helium (99.999% purity) is used as the carrier gas. Also, a purifier and an oxygen trap are incorporated into the analytical system as additional insurance against possible contamination.

6.2 Peak Separation Since the TCD exhibits universal responses and detects all gas components except the carrier (helium, in this case), the appropriate temperature program, column flow rates and column packing must be used in order to separate all of the permanent gases with an exception of argon

6.3 Argon In this method, argon (0.93% by volume in ambient air) is not chromatographically separated from oxygen; therefore, results are reported as oxygen/argon.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review and reporting per the corresponding standard operating procedures. Laboratory personnel that have successfully demonstrated the ability to generate acceptable results according to this SOP are approved to perform sample analysis and interpretation of the results.

7.2 The department supervisor/manager or designee shall perform final review and sign-off on the data.

7.3 Demonstration of Capability

Training demonstrations shall be conducted in accordance with the *SOP for Training Policy,* DoD QSM 5.0, and TNI requirements. An initial demonstration of proficiency must be performed prior to independent analyses of samples. In addition, ongoing demonstration must be performed annually.

Once performance is found to be acceptable, a certification statement must be completed by the QA Manager and either the immediate supervisor or Laboratory Director and retained on file as a demonstration of compliance.

7.3.1 Quarterly Demonstration A demonstration of method sensitivity must be performed *quarterly on each instrument* performing this method.

1) A spike at the current LOD must be analyzed if results are to be reported below the MRL.
2) Verification of precision and bias at the LOQ must be performed.

Refer to Section 12.4 (LOQ) and 12.11.1 (LOD) for additional information on how these demonstrations are to be performed as well as the acceptance criteria.

7.3.2 Annual Demonstration Each analyst must perform this demonstration both initially and annually. Analyze four LCS standards at 1-4x the MRL (LOQ) either concurrently or over a period of days as a verification of precision and bias of the quantitation range. The standard deviation (n-1) and average percent

recovery of the four replicates are compared against current laboratory control limits for precision and bias. See Attachment 4.

7.3.3 Change in Personnel, Instruments, Method and/or Matrix The requirements in Sections 7.3.1 and 7.3.2 must be performed per the schedule noted and when there is a change in personnel, instruments, method or matrix. "Change" refers to any change in personnel, instrument, test method, or sample matrix that potentially affects the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, or other components of the sample analytical system, or a method revision).

All attempts at this demonstration must be completed and turned into the QA department for retention. Once performance is found to be acceptable, a required certification statement will be completed by the QA Manager and either the immediate supervisor or Laboratory Director and retained on file as a demonstration of compliance.

8) Sample Collection, Handling, and Preservation

8.1 The samples are collected and delivered to the laboratory for analysis in either Tedlar bags, specially prepared canisters, or glass sampling bottles (Bottle Vac. Entech Instruments). Samples collected in bags must be analyzed within 72 hours after sample collection unless otherwise specified by the client. Samples delivered in cleaned, evacuated summa or other specially prepared containers do not have a specified holding time for atmospheric gases but this laboratory recommends that samples be analyzed within 30 days from the date of collection.

9) Equipment and Supplies

9.1 Gas Chromatograph The analysis is performed using a Hewlett-Packard model 5890 series II gas chromatograph or equivalent equipped with a thermal conductivity detector.

9.1.1 Column 6' x 1/8" stainless steel column packed with 60/80-mesh carbosphere.

Conditioning of the chromatographic column is required prior to use of the system. The column should be conditioned with a continuous flow of chromatographic grade Helium and temperature programmed from 35˚C to 200˚C at a rate of five degrees per minute. The column should be held at 200˚C for at least four hours.

9.1.2 Sample Loop Stainless steel tubing with a 1/16" diameter (various lengths).

9.1.3 Conditioning System The system is able to maintain the column and sample loop at a constant temperature.

9.2 Adsorption Tubes In addition to a thermal gas purifier incorporated into the system, an oxygen trap shall be utilized to remove any O2 from the carrier gas to help in extending the life of the TCD filaments.

9.3 Sampling Media Tedlar bags, Summa canisters, or glass bottles may be supplied to the client for sampling purposes. These samples are submitted to the laboratory for analysis. Summa canisters must be conditioned and certified in accordance with the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters*.

10) Standards and Reagents

10.1 All samples, standards, and media must be stored separately. The concentration, preparation and expiration date as well as analyst's initials must be identified on the standard label. Each standard must also be uniquely identified with a laboratory ID number.

All standard certificates shall be noted with the standard identification number, date received and initials of the receiving analyst. They must then be given to the quality assurance department where they will be maintained. For additional information on these and other requirements, refer to the *SOP for Handling Consumable Materials.*

10.2 Carrier and Calibration Standard Balance Gas

10.2.1 Helium UHP/ZERO (99.999%) or higher in purity

10.3 Standards DoD compliance requires that second source standards be obtained from a second manufacturer. The use of a standard from a second lot is acceptable only when one manufacturer of the standard exists.

10.3.1 Purchased Standards These standards must be stored in accordance with the requirements described in the *SOP for Handling Consumable Materials*. These standards must be stored at ambient temperatures for a period of up to 2 years or as recommended by the manufacturer.

10.3.1.1 Scott Specialty Gas or Equivalent

Compound	Concentration
Carbon dioxide	~5.00%
Carbon monoxide	~5.00%
Hydrogen	~4.00%
Methane	~4.00%
Nitrogen	~5.00%
Oxygen	~5.00%
Balance Gas: Helium	

Note: The concentrations of these standards will change with each purchase and the specific concentration of each compound will be denoted on the standard as well as the Certificate of Analysis and used in all calculations.

10.3.1.2 Matheson or Equivalent

Compound	Concentration
Carbon dioxide	~5.00%
Carbon monoxide	~5.00%
Hydrogen	~4.00%
Methane	~4.00%
Nitrogen	~5.00%
Oxygen	~5.00%
Balance Gas: Helium	

Note: The concentrations of these standards will change with each purchase and the specific concentration of each compound will be denoted on the standard as well as the Certificate of Analysis and used in all calculations.

10.3.1.3 AirGas or equivalent (Neat gas standards)

Compound	Concentration
Hydrogen	99.999%
Oxygen	99.999%
Nitrogen	99.999%
Methane	99.999%
Carbon Dioxide	99.999%

10.3.2 Ambient Air Ambient air is analyzed once per batch to assess injector performance.

11) Method Calibration

11.1 Initial Calibration

Record the detector temperatures, GC temperature program, standard concentrations, and sample loop volume. All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, each analyte name, analyst's initials, concentration and response, response factor. Refer to Section 16.4 for the acceptance criteria.

11.1.1 Analysis Guidelines

- Analyze differing concentrations covering the desired calibration range by utilizing different sample loops. The dynamic range may be amended as long as all documentation reflects the correct concentrations.
- An ICAL shall be performed at a minimum annually.

11.1.2 Initial Calibration Requirements

Once a set of ICAL standards is analyzed, the previous ICAL may no longer be used to analyze new samples and it must be archived. The only time an archived ICAL can be used thereafter is to review or re-evaluate samples(s) previously processed using that ICAL.

1. A minimum of 5 concentrations, must be used to calculate the calibration curve.
2. Highest concentration, together with the lowest concentration, defines the calibration curve.
3. Lowest concentration must be at or below the method reporting limit.
4. The initial calibration event may not be interrupted by maintenance.
5. Only one value per concentration may be used.
6. Analyze calibration standards from low to high concentration.
7. All ICAL analyses must be completed within 48 hours.
8. One injection per 5 points (2 per 6) may be re-analyzed to replace "bad" injection(s).
9. Point dropping policy:
    - The following are guidelines to follow if points are to be reviewed to determine the appropriateness of dropping a point or injection.
    - Lowest concentration must be at the MRL and may not be dropped unless another concentration is added to the upper end of the curve. This would in turn raise the MRL.
    - Points at the high end may be dropped but another concentration must be added and used in the calculation. The curve range must be noted.
    - Points must not be dropped from the "interior" of a curve unless there is an assignable cause* for doing so that affects many (if not all) the analytes in the calibration standard. If a calibration standard

is to be dropped from the interior of the curve, all the analytes in the calibration standard must be dropped from all the analytes' calibration curves.

- If a point or a calibration standard is dropped, the reason must be documented (and the results maintained with the documentation for the final ICAL).
- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 48 hours).
- Once the ICAL has been used to calculate and report sample results, it is not to be changed.

* Assignable causes include

- Standard preparation error
- Instrument malfunction (e.g., it quits acquiring in the middle of the analysis)
- Bad injection or purge

10. A set of concentrations for a calibration curve is in the following table (Attachment 5). However these concentrations might change due to the availability of the standards. Other concentrations can be used as long as all other guidelines for the analysis of initial calibration are followed.

Note: Hydrogen may not be linear; therefore, if an average response factor or linear regression cannot be used, a quadratic curve fit may be employed. A quadratic (second order) model requires a minimum of five calibration points.

11.1.3 ICAL Update Procedure

1. Open most recent method.
2. Save to new ICAL method ID. The date used in method ID is the date files were analyzed.
3. Clear all responses prior to update initiation and/or clear levels if different concentrations are to be used (Initial Calibration → Clear All Calibration Responses; Initial Calibration → Clear All Calibration Levels).
4. Quantitate standard
5. Review all peaks for retention time, integration, etc.
6. Update responses for standard
7. Repeat for all standards
8. If necessary load midpoint standard and update retention times.
9. Save method.
10. Verify Calibration Files listed on Response Factor Report are correct (Both Primary and Secondary Reviewer).
11. Verify responses of Page 3 of Edit Compounds are correct (Both Primary and Secondary Reviewer).
12. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report (Both Primary and Secondary Reviewer).
13. Save Method. Confirm that no other copies of the method are open on other computer workstations.

Note: It is also acceptable to quantitate all standards and review all peaks before updating responses but steps 1-2 still must be completed initially. Step 3 also must be done prior to beginning ICAL update.

11.1.4 Initial Calibration Review

The ICAL checklist is used to document the review and approval process. The Analyst's calculation and assessment along with a peer review of all ICAL data

and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples.

11.1.5 Initial Calibration File

An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- Blank analysis quantitation report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Plot for quadratic fit for hydrogen, if necessary
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and evaluate continuing calibration report (aka Percent Difference Report)
- Injection log (optional)

11.1.6 Initial Calibration Verification Verify the initial calibration by analyzing an independent calibration verification standard (ICV). Utilize the standard described in Section 10.3.1 for the analysis of a second source standard. Refer to Section 16.5 for acceptance criteria.

12) Sample Preparation/Analysis

12.1 Analytical Sequence The analytical batch must be completed for the analysis of ≤20 field samples.

Analytical Sequence Guideline[1]

Sample Description (w/ICAL)	*Sample Description*
Calibration Stds.[2]	CCV[3]
ICV[4]	MB[5]
MB[5]	Lab Air[6]
Lab Air[6]	Samples 1-10[7]
Samples 1-10[7]	CCV[3]
CCV[3]	Samples 11-19[7]
Samples 11-19[7]	LD[8]
LD[8]	LCS[9]
CCV[3]	CCV[3]

[1]The batch QC may be analyzed in an order other than the one listed in this document; the analytical sequence specified below is a guideline.
[2]The initial calibration must be generated in accordance with the guidelines detailed in Section 11.1.1 of this document.
[3]In cases, where the ICAL is not performed the analytical sequence must begin with the analysis of a CCV standard. In an external standard calibration the CCV is to be analyzed no less frequently than every ten samples or every 12 hours, whichever is more frequent, and the analytical sequence is to end with the analysis of a CCV standard.
[4]Every ICAL must be followed by a second source standard (ICV) which contains all of the target analytes. Same source as LCS; therefore, LCS is not required to be analyzed again.
[5]The method blank must be carried throughout the entire analytical process and be analyzed prior to any samples within the sequence. A method blank (MB) shall be run to monitor for laboratory introduced contamination.
[6]A volume of laboratory ambient air shall be analyzed at a rate of one per twenty sample injections or fewer.

[7]EPA Method 3C requires a duplicate injection for each sample. If the samples are being analyzed per a modified Method 3C, they are to be injected once (refer to note number 8). ASTM D 1946 requires only a single injection.

[8]Every batch must include the analysis of a laboratory duplicate. Samples selected for duplicate analysis shall be rotated among client samples. In addition, if performing EPA Method 3C without modification (duplicate injection), the laboratory duplicate analysis will not be necessary. A laboratory duplicate is considered a sample.

[9] A second source standard similar to 10.3.1.1 shall be analyzed once per twenty sample injections or fewer.

12.2 Conditions

The column and detector temperatures should be adjusted to the recommended levels. The column should be conditioned as instructed in Section 9.1.1. Once the GC/TCD system is optimized for analytical separation and sensitivity, the identical sample operating conditions must be used to analyze all samples, blanks, calibration standards and quality control samples.

The recommended settings and system parameters for GC01 are as follows:

Sample Inlet: GC
Injection Source: Sample Loop
Run Time: ~8 min

OVEN

Initial Temperature: 50˚C *Maximum Temperature*: 250˚C
Initial Time: 2.0 min *Equilibration Time*: 0.0 min

Ramps: Rate: 30˚/min
Final Temp.: 200˚C
Final Time: 1 min

COLUMN

Type: Packed
Model: Carbosphere 60/80
Dimensions: 6’ x 1/8”

DETECTOR

Temperature: 260˚C
Reference Flow: 45mL/min
He Make up: 20mL/min

The recommended settings and system parameters for GC20 are as follows:

Sample Inlet: GC
Injection Source: Sample Loop
Run Time: ~6.5 min

OVEN

Initial Temperature: 50˚C *Maximum Temperature*: 250˚C
Initial Time: 1.0 min *Equilibration Time*: 0.0 min

Ramps: Rate: 30˚/min
Final Temp.: 200˚C
Final Time: 0.5 min

COLUMN

Type: Packed
Model: shin carbon ST 100/120
Dimensions: 2 meters 1mm ID

DETECTOR

Temperature: 300˚C
Reference Flow: 20mL/min
He Make up: 2mL/min

12.3 Retention Time (RT) Windows

Retention time windows for each target analyte must be generated whenever there is a major change in instrument conditions including flow rates or when standard analyses result in analyte retention times outside the established windows. The procedure for determining the retention time windows for this method is as follows. However, other approaches may be employed, providing that the analyst can demonstrate that they provide performance appropriate for the intended application. For example, the analyst may use the corresponding retention times from the initial calibration as they may show shifts in RTs due to the volume injected (higher concentrations lead to wider peaks).

1. Make sure that the system is operating reliably and that the system conditions have been optimized for the target analytes in the sample matrix to be analyzed.
2. Make four injections of all applicable standard mixes over a 72 hour period. Make the injections cover the entire 72-hour period or the end result could be windows, which are too tight.
3. Record the retention time for each single component analyte to three decimal places. Calculate the mean and standard deviation of the four absolute retention times for each single component analyte and surrogate
4. If the standard deviation of the retention times for the target compound is 0.000, then additional injections may be included or the use of a default standard deviation of 0.01 minutes.
5. The width of the retention time window for each analyte is defined as ±3 times the standard deviation of the mean absolute retention time established during the 72 hour period. If the default standard deviation of 0.01 is used, the width of the window will be 0.03 minutes.
6. Establish the center of the retention time window for each analyte by using the absolute retention time for each analyte from the continuing calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Retention time windows must be calculated for each analyte on each instrument. New retention time windows must be established when a new column is installed.

12.4 LOQ Establishment, Verification, and Acceptance Criteria

A) The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
B) The LOQ for each analyte must be ≥ the analyte's LOD.
C) Initially a passing demonstration of precision and bias must be performed at the LOQ.
D) Run CCV 2 times at LOQ and:
   1) Evaluate the LOQ for precision and bias using current control chart limits.
   2) Check the signal to noise ratio (S/N) using the software. The S/N ratio must be at least 3:1 for each analyte.
E) If anything fails, verify at higher level and notify reporting. Also, make a note in the ICAL documentation.
F) Turn in all LOQ verification data (quant reports and software reports/checks) to QA (regardless of pass/fail).
G) Verify the LOQ on each instrument quarterly by running the CCV at the LOQ and verifying that ongoing precision and bias requirements are met.

12.5 Continuing Calibration Verification

A continuing calibration check shall be performed at the beginning and end of an analytical sequence and every ten field samples, not to exceed a 12 hour period. The concentration of the calibration verification may be varied within the established calibration range. Refer to Section 16.6 for acceptance criteria.

12.6 Laboratory Control Sample

A second source standard similar to Section 10.3.1.1 shall be analyzed once per closed batch. Refer to Section 16.11 for acceptance criteria.

12.7 Method Blank

A method blank must be analyzed by sampling chromatographic grade helium. Refer to Section 16.8 for acceptance criteria.

12.8 Sample Analysis

Refer to Section 16.10 for the acceptance criteria.

12.8.1 Container Pressurization Sample analysis must be made using the same instrument parameters as that of the calibration standards. Refer to the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters* for the procedure of how containers are to be pressurized prior to analysis. The analyst shall record the appropriate pressures on the Service Request form.

12.8.2 Sample Analysis Sample analysis is performed with the utilization of a sample loop equipped with a pump. If the sample container is not equipped with a sampling valve appropriate for this use, the sample container shall be fitted with an adapter. The dead volume within the adapter shall be evacuated and the sample loop flushed then filled with sample gas. Analyze each sample in duplicate (calculate the percent difference of the calculated concentration of each analysis) unless performing a single injection modification or referencing ASTM D 1946 (refer to Section 12.8.3, #2).

Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments). Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fitting higher than 80ºC.

12.8.3 Sample Re-analysis

1. If the response of any permanent gas analyte in a sample is greater than the response of that analyte in the ICAL (outside the ICAL upper calibration range) the sample shall be reanalyzed using a smaller loop.

   Dilution (i.e. Tedlar bags) would compromise sample integrity with the addition of laboratory air. Guidance in performing dilutions and exceptions to this requirement are given below.

   - The dilution factor chosen should keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument. Additional compounds may be reported as long as they are within the calibration range.

2. If the percent difference between the duplicate injection (analysis without modification) is greater than the acceptance criterion of 5%, the sample must be re-analyzed and repeated until acceptable *consecutive* numbers are achieved.

12.9 Laboratory Duplicate (LD)

If the method is being performed with a single injection modification, then the analysis of a LD is required to show precision. The laboratory duplicate should be rotated among clients, whenever possible. Refer to Section 16.9 for acceptance criteria.

12.10 Manual Integration

The integration for each peak is checked to ensure that it has been integrated properly. Assuming an incorrect automatic integration the analyst shall conduct the manual integration in accordance with the *SOP for Manual Integration Policy* including all documentation and reviews associated with the process. The review shall include the analyst and peer reviewer initialing and dating the manual integration as an indication of acceptability and approval.

12.11 Detection Limits and Limits of Detection

If results are to be reported below the MRL, an MDL study must be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* Method detection limits must be determined annually and each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation is such that it affects the sensitivity of the analysis. The MDL study shall be performed on each instrument for which this method is performed. All supporting data must be approved and retained.

The detection limit shall be used to determine the LOD for each analyte. Once determined on each instrument, the highest LOD (for each analyte from all instrument determinations) shall be used as the uniform LOD.

12.11.1 Performance and Acceptance Criteria

1. Perform Limit of Detection (LOD) verification on all instruments (performing this method) immediately following the MDL study. Spike the LOD at 2-4x the MDL; the spike level establishes the LOD.
2. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).
   - It is specific to each combination of analyte, matrix, method and instrument configuration.
   - The LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.
3. If the LOD verification fails (per #2), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.
4. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

Note: Per the DoD QSM 5.0 and TNI Standard, it is not necessary to perform a MDL study when results are not to be reported below the LOQ/MRL.

12.12 Ambient Air

An ambient laboratory air sample shall be analyzed once per closed batch (20 or fewer sample injections). Refer to Section 16.7 for the acceptance criteria and corrective action.

12.13 Method Modifications

12.13.1 The following are EPA 3C method modifications:

- Reporting carbon dioxide, methane, nitrogen, and oxygen from a single sample injection.
- Reporting hydrogen and carbon monoxide (these compounds are not included in 3C method).
- Sample results are normalized per ASTM D 1946.
- Use of sample containers other than backfilled Summa canisters.

12.13.2 The modification for ASTM D 1946 is the omission of ethane and ethane.

12.13.3 The column backflush procedure described in method ASTM D 1945-03 is not performed.

12.14 Loop calibration

The loop injection port has a standard loop of approximately 100ul to introduce sample to the instrument. There are other loops that are used to introduce smaller and larger amounts and these are calibrated against the normal loop for a known dilution factor.

12.14.1 Calibration Procedure

A standard of approximately 50000ppm for all analytes is analyzed three times with the normal loop. The area counts for all analytes with the exception of hydrogen are summed for each standard. This summation is averaged of the three standard injections. This procedure is duplicated using another loop. The dilution factor is the ratio of the average area counts of the normal loop divided by the average area counts of the other sampling loop.

For current Loop Ratios see Table 1.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Data System

Load the appropriate analytical sequence (e.g., J:\GC1\sequence\fxgs_25c.s). Enter the analytical sequence information in the table window, including sample/standard name. Load the appropriate quantitation analytical method (e.g., J:\gc1\methods\"appropriate ICAL"). Run the sequence and analyze the standards and samples in the order specified.

14.2 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. Files shall be named with a two-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files shall be saved in a unique sub-directory on the server. An example of how the analyst must store analytical data is as follows:

Instrument Number/Data/Method ID/yr_month/*.d

* Injection (automatically assigned based on order of injection)

14.3 Sufficient raw data records must be retained of the analysis, instrument calibrations and method detection limit studies. This includes analysis/calibration date, test method, instrument, sample identification, each analyte name, analyst's initials, concentration and response, and standards used for the analysis and calibrations as well as any manual integrations and all manual calculations including sample dilutions. All information entered and reported on the quantitation reports must be complete and accurate.

14.4 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time of analysis (both are required for Tedlar bags since the holding time is 72 hours), instrument operating conditions/parameters (or reference to such data), analysis type, manual integrations, all manual calculations, analyst's initials, sample preparation (pressure readings and balance gas), standard and reagent origin, sample receipt, calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, and assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 Initial Calibration

- Response Factor for each injection (equation number 5)
- Mean Response Factor using all injections (equation number 6)
- Percent Relative Standard Deviation (equation numbers 5,6,7, and 8)

Hydrogen (if quadractic is used):

- Coefficient of Determination (equation number 12)

15.2 Initial Calibration Verification

- Response Factor (equation number 5)
- Mean Area Response (equation number 6)
- Percent Difference (equation number 3)

15.3 Continuing Calibration Verification

- Response Factor (equation number 5)
- Mean Area Response, where necessary (equation number 6)
- Percent Difference (equation number 3)

15.4 Laboratory Duplicate and Method 3C without modification

- Relative Percent Difference (equation number 4)

15.5 Sample Analysis

Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.

All permanent gas results are normalized as dry gas to 99.99% proportionately, in order to reflect the true composition of the sample. It is the practice of the laboratory to normalize results of permanent gas analysis, except under special circumstances that occur where the normalization of the results is not utilized or the normalization procedure is modified. For example, samples containing greater than 0.01% by volume of measured constituents other than permanent gases (for instance high hydrocarbon or sulfur levels) are normalized to 99.99% minus the percent contribution from components other than permanent gases.

- Calculate the average area of the two injections, where necessary (equation number 2)
- Calculate the dilution factor, where necessary (equation number 1)
- Analyte concentration (equation number 9)
- Hydrogen concentration (equation number 14)
- Normalization (equation number 11)

When the analysis of a sample produces permanent gas results whereby the total is significantly less than expected, accounting for experimental error, it is the laboratory's practice to reanalyze the sample in question as well as the laboratory air. This will determine if there is a problem with the analytical system. If there is no problem with the system and the results are the same refer to the following example.

If the total of the permanent gas analysis is less than 60.0% by volume and the laboratory is not requested to perform additional analyses, the results would be reported unnormalized. The decisions whether to report the unnormalized results is at the discretion of the analyst and department supervisor.

15.6 Laboratory Control Sample

- Calculate the percent recovery (equation number 10)

15.7 Calculations

15.7.1 Equation Number 1

Dilution Factor

$$DF = \frac{V_{STD}}{V_S}$$

Where:

DF = dilution factor
V_{STD} = volume of standard loop
V_S = volume of sample loop

15.7.2 Equation Number 2

Average

$$\frac{x + y}{n}$$

where:

x = response from the first injection
y = response from the second consecutive injection
n = number being averaged together

15.7.3 Equation Number 3

Percent Difference, %D,

The %D is used for evaluating ICV and CCV vs. the initial calibration

$$\%D = \frac{C_{CCVorICV} - C_{std}}{C_{std}}(100)$$

where, for any given analyte:

$C_{CCVorICV}$ is the calculated concentration being evaluated
C_{std} is the concentration of the standard used

15.7.4 Equation Number 4

Relative Percent Difference (RPD)

$$\frac{|R_1 - R_2|}{\left(\frac{R_1 + R_2}{2}\right)} x100$$

where:

R_1 First measurement value
R_2 Second measurement value

15.7.5 Equation Number 5

Response Factor (RF)

The response factor, for analyte *x* is given by:

$$RF = \frac{A_x}{C_x}$$

where:

A_x = Area of the analyte in the standard
C_x = Concentration of the analyte in the standard

15.7.6 Equation Number 6

Average (or Mean) RF

$$\overline{RF} = \frac{\sum_{i=1}^{N} RF_i}{N}$$

where:
RF_i are the individual RFs from each injection in the initial calibration curve
N is the number of injections

15.7.7 Equation Number 7

Standard Deviation, SD:

$$SD = \sqrt{\sum_{i=1}^{N} \frac{\left(RF_i - \overline{RF}\right)^2}{N-1}}$$

where:

RF_i are the individual RFs from each concentration level in the initial calibration curve

$\overline{RF}$ Average (or Mean) RF of all injections in the initial calibration curve

N total number of injections

15.7.8 Equation Number 8

Percent Relative Standard Deviation, %RSD:

$$\%RSD = \frac{SD}{\overline{RF}}(100)$$

where:

SD Standard Deviation calculated in equation number 3

$\overline{RF}$ Average or Mean RF

15.7.9 Equation Number 9

Concentration (C):

$$C = \frac{Area}{\overline{RF}} \times \frac{D_{SLV}}{A_{SLV}}$$

or

$$C = \frac{\overline{Area}}{\overline{RF}} \times \frac{D_{SLV}}{A_{SLV}}$$

where:

Area is the area obtained from the chromatogram

$\overline{Area}$ Mean area for both injections, if performing analysis without modification

$\overline{RF}$ Average (or Mean) RF of all concentration levels in the initial calibration curve

D_{SLV} default sample loop volume

A_{SLV} actual sample loop volume

15.7.10 Equation Number 10

Percent Recovery (%R):

$$\%R = \frac{C}{S} x100$$

where:

C = Concentration of the analyte recovered
S = Spiked amount

15.7.11 Equation Number 11

Normalization
Divide each analyte's calculated concentration (percent) by the percent sum of the permanent gases in the sample and multiply by 99.99 or the adjusted value.

15.7.12 Equation Number 12

Quadratic (Coefficient of Determination)

$$COD = \frac{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right)\sum_{i=1}^{n}(y_{obs} - Y_i)^2}{\sum_{i=1}^{n}\left(y_{obs} - \bar{y}\right)^2}$$

where:

y_{obs} = Observed response (area) for each concentration from each initial calibration standard

$\bar{y}$ = Mean observed response from the initial calibration

Y_i = Calculated response at each concentration from the initial calibration

n = Total number of injections

p = Number of adjustable parameters in the polynomial equation (i.e., 3 for a third order; 2 for a second order polynomial)

15.7.13 Equation Number 13

Quadratic Fit

$R = AX^2 + BX + C$

where:

R = response
X = quantity, ng
A, B and C = are coefficients in the equation

15.7.14 Equation Number 14

Analyte Concentration (using equation number 13)

$$X = \frac{\sqrt{4A(R-C) + B^2} - B}{2A}$$

15.8 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated following the data review checklist in Attachment 3. The

data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The data review checklist shall be used to document the review process. Once it has been completed, the checklist must be initialed, dated and filed with each job file. Results must not be reported until after they are appropriately reviewed according to this SOP, the *SOP for Data Review and Reporting* and the *SOP for Laboratory Ethics and Data Integrity*.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.5.

15.9 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and all information required by this SOP and the *SOP for Data Review and Reporting*. The following are situations whereby the results shall be reported as being analyzed by Modified EPA Method 3C: single injection, reporting hydrogen and carbon monoxide and if analyzing replicate injections (for 3C without modification) and the samples are submitted in Tedlar bags.

15.9.1 EPA Method 3C Modifications

- Single injection
- Sample container other than backfilled Summa canisters
- Reporting carbon monoxide and /or hydrogen

15.10 Sample Preparation and Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis, and Tracking* must be generated when there are any specific sample composition information, sample preparation, analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved.

This form is necessary as a means for documenting any unusual or noncompliant information. This form, among other information, will be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through preparation, analysis, data reduction, and reporting.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 This section of the standard operating procedure contains technical acceptance criteria and preferred corrective actions to data nonconformities. Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate.

16.2 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.3 It must be determined if there are any instrumentation problems contributing to out of control QC data and the analyst must determine if this has affected sample results. This being the case, all samples (including QC) that are affected by instrumentation problems must be re-analyzed following any necessary maintenance activity.

16.4 Initial Calibration

16.4.1 Acceptance Criteria

- If a quadratic fit (for hydrogen) is used it should be forced through zero.
- The percent relative standard deviation (%RSD) for the response factors must be ≤15% for all compounds except hydrogen if utilizing a quadratic curve.
- Hydrogen may be fitted to a quadratic curve where the coefficient of determination (COD) shall be ≥0.99.
- The retention time for each point must within 0.06 minutes of the mean RT. However it must be noted that higher injection volumes and/or higher concentrations of any analyte may not meet this criteria, which is acceptable.

16.4.2 Corrective Action

If the initial calibration technical acceptance criteria are not met, inspect the system for possible sources. Check standards and re-analyze (per ICAL policy in Section 11.1.2), if necessary. Also, it may be necessary to perform maintenance or perform other corrective actions to meet the technical acceptance criteria. Attempt another initial calibration and make a notation in the maintenance logbook regarding any maintenance steps taken. If the recalibration does not meet the established criteria, new calibration standards must be made. A demonstration of an in-control system is required before proceeding with the analysis.

16.5 Initial Calibration Verification (ICV) Standard

16.5.1 Acceptance Criteria

- The percent difference for each compound in the ICV must be ≤15%.

16.5.2 Corrective Action

If the ICV does not pass the criteria the standard must be reanalyzed and reevaluated. If reanalysis also fails to produce an acceptable recovery, documented corrective action must be initiated. This may include instrument maintenance, a new ICV standard or the analysis of a new initial calibration curve.

16.6 Continuing Calibration Verification (CCV) Standard

16.6.1 Acceptance Criteria

- The percent difference for each analyte in the CCV must be ≤10%, except hydrogen which must be ≤15%.
- The retention time for each analyte in the standard must be within 0.33minutes of the mean RT (of the corresponding analyte) from the ICAL.

16.6.2 Corrective Action

If the continuing calibration fails to meet expected criterion, the CCV may be reanalyzed (no more than two runs of the CCV standard may be analyzed without documented corrective action, i.e. a notation in the logbook). If the acceptance criterion is still not met, it may be necessary to perform maintenance prior to reanalysis. If routine maintenance does not correct the problem, a new initial calibration must be performed on the instrument.

If the retention time criterion is not met, leak check the system, check the carrier gas cylinders, determine if there has been a loss of pressure in lines. If the analytes do not fall within the generated windows, a new retention time window should be generated.

DoD QSM 5.0 Requirement: If a CCV fails, the laboratory must immediately analyze two additional consecutive CCVs (immediately is defined as within one hour).

- Both of these CCVs must meet acceptance criteria in order for samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

16.7 Ambient Air

16.7.1 Acceptance Criteria

- The sum of the results for nitrogen and oxygen/argon must fall between 90% and 110% (un-normalized).

16.7.2 Corrective Action

Reanalyze the lab ambient air and if the results still do not meet the criterion, the sample line should be purged with nitrogen to release any blockage. This is particularly important if the results for the first criterion are low. Also, if the result is low the system should be checked for leaks. All standards, samples and QC samples associated with the lab ambient air should be reanalyzed following the maintenance activity if it is determined that the results could have been affected.

16.8 Method Blank

16.8.1 Acceptance Criteria

- The method blank result for any target analyte must not be greater than the method reporting limit. Also, the blank should not contain additional compounds with elution characteristics that would interfere with identification and measurement of a target analyte.
- For DoD samples, the method blank will be considered to be contaminated if:
  1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
  2. The concentration of any common laboratory contaminant in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
  3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

  The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

16.8.2 Corrective Action

Re-inject the method blank and if the results are the same, analyze an instrument blank (inject without turning on the pump) to determine if the contamination is the blank canister or the analytical system. Corrective action documentation must be initiated following a failed second analysis. If the

system is contaminated, then both the method blank(s) and the associated samples in question must be re-analyzed.

16.9 Laboratory Duplicates (Modified EPA Method 3C)

16.9.1 Acceptance Criteria

- Every batch of twenty or fewer samples, if performing EPA Method 3C with modification, must include the analysis of a laboratory duplicate as a measurement of method precision. Refer to Attachment 4 of this document.

16.9.2 Corrective Action

If the replicate results do not fall within the technical acceptance window, the sample should be re-analyzed. If the results are still unacceptable and there does not appear to be any matrix effects, interfering peaks, or instrument problems, the results for both injections shall be reported to the client with the appropriate qualifier.

16.10 Sample Analysis

16.10.1 Acceptance Criteria

- Samples out of holding time must be handled according to Section 16.12.
- The sample replicate injections are acceptable when the RPD is within ±5% (analysis without modification must consist of consecutive injections).
- Analyte retention time must be within the daily RT window and within 0.33minutes of the mean RT in the ICAL.

16.10.2 Corrective Action

Analysis Without Modification If the two injections do not agree, run additional samples until consistent area data are obtained in two consecutive injections.

Analysis With or Without Modification If the retention time for any analyte falls outside of the retention time window from the latest daily calibration or average initial calibration retention time, the system must be inspected for a change in the head pressure and the results evaluated and reported accordingly.

Results not bracketed by initial instrument calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags.

16.11 Laboratory Control Sample (LCS)

16.11.1 Acceptance Criteria

- The percent recovery must fall within the fixed recoveries of 85-115% or laboratory generated control limits when available. Refer to Attachment D.

16.11.2 Corrective Action

If the LCS criteria are not met, determine whether the cause is instrumentation problems, result of poor injection or a poor LCS. If necessary perform maintenance, re-inject the LCS or make a new standard. If the LCS criteria are still not met, a new ICAL must be run or the data must be qualified.

16.12 Expired Sample Holding Time

The customer shall be notified by the Project Manager (best attempt) when informed by an Analyst, Team Lead or SMO that the sample's holding time was missed. The customer must decide if the sample analysis shall continue. The documentation of missed holding time and the client's decision to proceed must be included in the

corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s) as detailed in Appendix D of the most current Quality Assurance Manual.

18.2 When analysis quality control results are unacceptable:

If the associated samples are within holding time, re-analyze the sample with criteria under control. Alternatively, evaluate the effect on the sample results and report the results with qualifiers and/or discuss in the case narrative if the effect is judged insignificant.

18.2.1 Method Blank If an analyte in the method blank is found to be unacceptable and the analyte is also found in associated samples, those sample results shall be "flagged" in the report. If the analyte is found in the blank but not in the sample and all other quality control meets acceptance criteria then the results for the sample may be reported without a qualifier. However, if other QC is out of control then an evaluation must be made and the results reported accordingly.

18.2.2 Laboratory Duplicate (Analysis with Modification) If the results from the reanalysis are unacceptable, and there does not appear to be any matrix effects, interfering peaks, or instrument problems, the results for both injections shall be reported to the client. In addition, other results from the same analytical sequence should be reported with the appropriate qualifier.

18.2.3 Laboratory Control Sample An unacceptable LCS must be evaluated along with the sample analysis and reported accordingly.

18.2.4 Initial Calibration Sample data may NOT be reported with an unacceptable ICAL.

18.2.5 CCV Sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special condition:

When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the sample affected by the unacceptable CCV shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

18.3 Sample Out of Control

18.3.1 Hold Time All Tedlar bag samples analyzed outside of the required hold time of 72 hours must be reported with the appropriate qualifier.

18.3.2 Retention Time All analytes outside of the retention time window (following a retention time evaluation) must be reported with the appropriate qualitative uncertainty, where necessary.

18.3.3 Duplicate Results (Analysis without modification) If the results from any of the repeated injections are still unacceptable (and other sample results were acceptable), and there does not appear to be any matrix effects, interfering peaks, or instrument problems, the results for both injections shall be reported to the client. If the out-of-control results are due to matrix interferences, report the results with a matrix interference qualifier.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

19.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. MDLs can be obtained using standards at a concentration of about 300ppm to 1000ppm and making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence.

The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. Refer to Section 12.11.1 for the LOD verification criteria.

Note: Per the DoD QSM 5.0 and TNI Standard, it is not necessary to perform a MDL study when results are not to be reported below the LOQ/MRL.

19.3 Accuracy and Precision

Refer to Section 16.9 for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 15%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in attachment 4. Refer to Section 12.4 for the accuracy and precision LOQ requirements.

19.4 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Ongoing demonstration of capability shall be performed and documented; however, the initial demonstration of method capability is not required.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
13.0	12/31/15	C. Humphrey	7.3 – Removed reference to NELAC
			12.11.1 – Removed reference to NELAC
			12.13.1 – Revised to add clarification to EPA 3C method modifications
			19.2 – Removed reference to NELAC
			Attachment 4 – Updated control limits

21) References and Related Documents

21.1 "*Determination of Carbon Dioxide, Methane, Nitrogen, and Oxygen from Stationary Sources*", EPA Method 3C

21.2 ASTM D 1946-90 (Reapproved 2006), "*Standard Practice for Analysis of Reformed Gas by Gas Chromatography*".

21.3 ASTM D 1945-03 (Reapproved 2010), "*Standard Test Method for Analysis of Natural Gas by Gas Chromatography".*

21.4 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.

21.5 *SOP for Batches and Sequences*, SOP ID ADM-BATCH_SEQ

21.6 *SOP for Making Entries onto Analytical Records*, SOP ID CE-QA007

21.7 *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*, SOP ID CE-QA011

21.8 *SOP for Manual Integration Policy*, SOP ID CE-QA002

21.9 *SOP for Nonconformance and Corrective Action*, SOP ID CE-QA008

22) Appendix

22.1 Tables

Table 1 – Loop Ratios

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Data Review Checklist

Attachment 4 – MRLs and Control Limits

Attachment 5 – Calibration Curve Concentrations

Table 1

Loop Ratios	
Normal Loop	1.00
Small Loop	0.1556
Medium Loop 1	0.4202
Medium Loop 2	0.8521
Large Loop	1.280

Note: New loop ratios may be established prior to the revision of this document, refer to the most recent loop ratios.

Attachment 1

Training Plan

Training Plan for Analysis of Fixed Gases by GC/TCD

Trainee _____ Trainer _____ Instrument _____

1. Read SOP Trainer ____ Trainee ____ Date _____
2. Read Method: EPA Method 3C, ASTM D 1946, ASTM D 1945 Trainer ____ Trainee ____ Date _____
3. Demonstrated understanding of the scientific basis of the analysis
   Gas chromatography Thermal Conductivity Detector Trainer ____ Trainee ____ Date _____
4. Demonstrated familiarity with related SOPs Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences
   SOP for Making Entries onto Analytical Records
   SOP for Manual Integration Policy
   SOP for Significant Figures
   SOP for Nonconformance and Corrective Action
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation
5. Observe performance of SOP Trainer ____ Trainee ____ Date _____
   ___standard preparation
   ___sample preparation (gas-phase dilutions)
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant introduction
   ___data reduction and reporting
6. Perform SOP with supervision Trainer ____ Trainee ____ Date _____
   ___standard preparation
   ___sample preparation (gas-phase dilutions)
   ___analytical sequence setup
   ___initial calibration and initial calibration verification
   ___continuing calibration verification
   ___sample analysis
   ___EnviroQuant use
   ___data reduction and reporting
7. Independent performance of the SOP Trainer ____ Trainee ____ Date _____
   ___standard preparation
   ___sample preparation (gas-phase dilutions)
   ___analytical sequence setup
   ___initial calibration and continuing calibration verification
   ___sample analysis
   ___EnviroQuant proficiency
   ___data reduction and reporting
   ___initial demonstration of competency
      ___ Four consecutive laboratory control samples
8. Instrument operation and maintenance Trainer ____ Trainee ____ Date _____
   ___gas chromatograph and column installation (packed)
   ___detector (TCD) setup and maintenance
   ___data system

Attachment 2

Initial Calibration Checklist

Initial Calibration Checklist (Fixed Gases)

Analysis: EPA Method 3C / ASTM D 1946 / ASTM D 1945

ICAL Date _____ Instrument ☐ GC01 ☐ GC ____

Analyst		Reviewer
☐	1. Is the required documentation in the ICAL file?	☐
	☐ Sequence report	
	☐ Blank analysis Quantitation Report	
	☐ Calibration Status Report (aka Calibration History) – Initial	
	☐ Response Factor Report and Plot for Hydrogen	
	☐ Quantitation Report for each calibration standard (including manual integration documentation – before and after printouts)	
	☐ ICV Quantitation Report and Evaluate Continuing Calibration Report (aka Percent Diff. report)	
☐	2. Was the ICAL performed continuously (i.e., not interrupted for maintenance or sample analysis)?	☐
☐	3. All the calibration standards analyzed within 48 hours of each other?	☐
☐	4. Were the standards analyzed from low concentration to high concentration?	☐
☐	5. Are all the analytes in the blank analysis < MRL?	☐
☐	6. Does each analyte's ICAL include a minimum of 5 consecutive concentrations?	☐
☐	7. Was each standard concentration included in the ICAL?	☐
☐	8. If a point is dropped, is information noted in the ICAL explaining the reason?	☐
☐	9. Does this follow the Laboratory's point dropping policy? Are the injections dropped for that concentration for each analyte?	☐
☐	10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL?	☐
☐	11. For each analyte, are there no levels skipped?	☐
☐	12. For analytes calibrated using RF, is the RSD ≤15%? For Hydrogen ≥0.99?	☐
☐	13. For the ICV analysis, is the percent recovery for each analyte 85-115%?	☐
☐	14. Are all peak integrations including manual integrations (per *SOP for Manual Integration Policy*) acceptable? *If so, initial and date the appropriate pages.*	☐

COMMENTS:

_____

_____

_____

_____

Analyst _____ Secondary Reviewer _____

Date _____ Date _____

Attachment 3

Data Review Checklist

Fixed Gases per EPA Method 3C / ASTM D 1946 / ASTM D 1945
Data Review Checklist
(Note exceptions and include Sample Preparation and Analysis Observations / Case Narrative Summary Form as appropriate)

Analysis Date _____ Instrument ☐ GC19 ☐ GC ____

Client _____ QC Level _____

Project # _____ Due Date _____

Modification ☐ Yes ☐ No

Analyst **Reviewer**

Initial Calibration

☐ 1. Is the referenced ICAL the most recent ICAL performed? ..... NA

☐ 2. Has the referenced ICAL been peer reviewed and all associated documentation including the ICAL review checklist available for review? ..... NA

☐ 3. Were all associated requirements within the specified limits? ..... NA

Data

☐ 1. Is the sample data documentation present and correct? ..... ☐
- ☐ Sample raw data?
- ☐ All target analyte responses within calibration range?
- ☐ All peak integration acceptable?
- ☐ All manual integrations flagged and documented (before and after)? If so, initial and date.
- ☐ All analyte retention times within the generated RT window?
- ☐ All calculations correct?
- ☐ First quantitation report initialed and dated by analyst?

☐ 2. Do all sample duplicate injections (if analyzing without modification have a RSD ≤5%? ..... ☐

☐ 3. CCV have a percent difference of ≤10% (≤15% for hydrogen)? ..... ☐

☐ 4. Is the retention time (for CCV) for each analyte in the standard within 0.33min from the mean RT (of the corresponding analyte) from the ICAL? ..... ☐

☐ 5. Is the sum of the gases in the lab air within 90% and 110%? ..... ☐

☐ 6. Are the %R for the LCS within the acceptance criteria for each analyte? ..... ☐

☐ 7. Are the analytes in the MB < MRL? ..... ☐

☐ 8. Do all reported analytes fall within the generated retention time windows? If not, is the reason for reporting analyte in the sample documented? ..... ☐

☐ 9. Is the RPD (with modification) for the LD within the laboratory generated RPD limits? ..... ☐

☐ 10. **DOD: Are manual integrations notated in the case narrative**? ..... ☐

COMMENTS:

_____

_____

_____

☐ LIMS Run Approval ☐ LIMS Supervisor Approval

Analyst _____ Secondary Reviewer _____

Date _____ Date _____

Attachment 4

Method Reporting Limits and Control Limits

Target Analytes with Associated MRLs

Compound	Method Reporting Limit
Hydrogen	1000ppm
Oxygen	1000ppm
Nitrogen	1000ppm
Carbon monoxide	1000ppm
Methane	1000ppm
Carbon dioxide	1000ppm

Laboratory Generated Control Limits – ASTM D 1946-90 / Modified EPA 3C Single Injection

Analyte	LCS - LCL (%R)	LCS - UCL (%R)	LD (RPD)
Hydrogen – H_2	83	114	16
Oxygen – O_2	84	121	16
Nitrogen – N_2	88	122	21
Carbon monoxide - CO	87	118	16
Methane – CH_4	85	116	16
Carbon dioxide – CO_2	84	117	16

Note: New limits may be established prior to the revision of this document, refer to the most recent control limits.

Attachment 5

Calibration Curve Concentrations

Suggested Calibration Curve Concentrations (ppm unless noted as %)

ICAL	Hydrogen	Oxygen	Nitrogen	Carbon Monoxide	Methane	Carbon Dioxide
1	373.69	467.11	466.18	470.85	373.69	467.11
2	2000	2500	2495	2520	2000	2500
3	7473.77	9342.21	9342.21	9379.58	7511.14	9323.53
4	40000	50000	50000	50200	40200	49900
5	467731.47	584664.34	584664.34	587002.99	470070.13	583495
6	99.999%					
7		99.999%				
8			99.999%			
9					99.999%	
10						99.999%

ICAL	Amount of Standard Spiked onto Instrument
1	small loop injection of a 2500ppm/2000ppm standard [1,2]
2	standard loop injection of a 2500ppm/2000ppm standard [1,2]
3	small loop injection of a purchased 5%/4% standard (see section 10.3.1.1) [2]
4	standard loop injection of a purchased 5%/4% standard (see section 10.3.1.1) [2]
5	large loop injection of a purchased 5%/4% standard (see section 10.3.1.1) [2]
6 through 10	standard loop injection of neat gas compounds (see section 10.3.1.1)

[1]2500ppm/2000ppm standard is made by introducing 600ml of a purchased 5%/4% standard into a 6 liter summa canister and pressurized to +14.7psig (29.4psi) with helium.
[2]The loop injection volumes are calculated as described in section 12.14 and shown in Table 1.

Calibration Range	
Hydrogen	1000ppm – 99.999%
Oxygen	1000ppm – 99.999%
Nitrogen	1000ppm – 99.999%
Carbon Monoxide	1000ppm – 58.700%
Methane	1000ppm – 99.999%
Carbon Dioxide	1000ppm – 99.999%

ALS Standard Operating Procedure

Proprietary - Uncontrolled Copy

DOCUMENT TITLE:	DETERMINATION OF AIR-PHASE PETROLEUM HYDROCARBONS (APH) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCED METHOD:	MADEP APH
SOP ID:	VOA-MAPH
REV. NUMBER:	09.0
EFFECTIVE DATE:	03/21/2015

ALS Environmental

STANDARD OPERATING PROCEDURE

DETERMINATION OF AIR-PHASE PETROLEUM HYDROCARBONS (APH) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

MADEP APH

SOP ID:	VOA-MAPH	Rev. Number:	09.0	Effective Date:	03/21/2015

Approved By: _____ Date: 3/10/15
Technical Manager (Volatile GC/MS) - Chris Parnell

Approved By: _____ Date: 3/10/15
QA Manager - Chaney Humphrey

Approved By: _____ Date: 3/11/15
Laboratory Director - Kelly Horiuchi

Archival Date:	_____	Doc Control ID#:	Non-Controlled	Editor:	_____

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DETERMINATION OF AIR-PHASE PETROLEUM HYDROCARBONS (APH) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1) Scope and Applicability

1.1 This procedure is based on and incorporates the requirements detailed in the Method for the Determination of Air-Phase Petroleum Hydrocarbons (APH), Revision 1, December 2009, Massachusetts Department of Environmental Protection. It is designed to measure the gaseous-phase concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in air. Volatile aliphatic hydrocarbons are collectively quantitated within two carbon number ranges: C5 through C8 and C9 through C12. In addition, volatile aromatic hydrocarbons are collectively quantitated within the C9-C10 range. Also, this method may be used to measure the individual concentrations of target APH analytes 1,3-butadiene, methyl-tert-butyl ether (MtBE), benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, and naphthalene in air. An extended list of target analytes also may be reported since this method overlaps with EPA Method TO-15.

1.2 This method typically applies to whole air samples received in Summa stainless steel canisters, with subsequent analysis by gas chromatography/mass spectrometry (GC/MS). The method reporting limit (MRL) for this method for each of the collective aliphatic and aromatic fractional ranges is approximately 2.5 – 20ug/m3. The MRL for the target APH analytes is compound specific but is approximately 0.50ug/m3. Refer to the most recent method detection limit study and initial calibration for the corresponding method detection and reporting limits. The reported MRL may be adjusted higher; however, the capability of achieving lower MRLs for specific project requirements must be thoroughly demonstrated and documented. The number of samples that may be analyzed in a 24-hour period is about twenty.

2) Summary of Procedure

2.1 Samples are collected in pre-cleaned, evacuated Summa stainless steel canisters. An aliquot of an air sample is concentrated on a solid adsorbent trap to collect the analytes of interest. To remove co-collected water vapor, the concentrated sample then goes through a water removal (dry purge) step. After the sample is pre-concentrated on a trap, the trap is heated and the APHs are thermally desorbed onto a refocusing cold trap. The APHs are then thermally desorbed onto the head of a capillary column once the cold trap is heated. The oven temperature (programmed) increases and the APHs elute and are detected by the mass spectrometer. The GC/MS utilizes a linear quadrupole system, which allows for it to be operated by either continuously scanning a wide range of mass to charge ratios (SCAN mode) or by Select Ion Monitoring mode (SIM), which consists of monitoring a small number of ions from a specified compound list.

2.2 Target APH analytes are identified and quantitated using characteristic ions. Collective concentrations of C9-C10 aromatic hydrocarbons are quantitated using extracted ions. Collective concentrations of aliphatic hydrocarbons fractions are quantitated using a total ion chromatogram, subtracting out target APH analytes and C9-C10 aromatic hydrocarbons. The target analytes will be quantitated and reported using EPA method TO-15. Since the sample pre-concentration steps and analytical conditions are identical for TO-15 and the Massachusetts APH method, all sample results can be generated from the same analytical run.

3) Definitions

3.1 Cryogen A refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Liquid nitrogen (cryogen) is used for this purpose and it has a boiling point of -195.8°C.

3.2 Gauge Pressure Pressure measure with reference to the surrounding atmospheric pressure, usually expressed in units of psi. Zero gauge pressure is equal to atmospheric (barometric) pressure.

3.3 MS-SCAN Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

3.4 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.5 Stock Standard A purchased, multi-component gas-phase mixture having certified concentrations, used to prepare working calibration standards.

3.6 Working Calibration Standard A gas-phase mixture of all the target analytes at a known concentration prepared by diluting a gas-phase stock standard into a Summa canister. Used for calibrations. Standard canisters prepared from methanol stocks are not allowed.

3.7 Calibration or Standard Curve A calibration or standard curve is a graph which plots the concentration of a compound (or an analyte) versus the instrument response to the compound.

3.8 Initial Calibration Verification (ICV) Standard A gas-phase standard prepared in the laboratory containing known concentration(s) of analytes of interest. It is prepared from gas-phase stock standards which are from a different source than the standards used to prepare the working calibration standards. Standard canisters prepared from methanol stocks are not allowed.

3.9 Continuing Calibration Verification (CCV) Standard A working calibration standard which is analyzed at specific intervals in order to verify that the instrument continues to meet the calibration criteria.

3.10 Field Sample A sample collected and delivered to the laboratory for analysis.

3.11 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop "ticks" have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.12 Batch Quality Control (QC) Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD).

3.13 Internal Standard Calibration Compares the instrument responses from the target compound in the sample to the responses of specific standards (called internal standards), which are added to the sample or sample preparation prior to analysis. The ratio of the peak area (or height) of the target compound in the sample or sample preparation is compared to a similar ratio derived for each calibration standard.

3.14 May This action, activity, or procedural step is neither required nor prohibited.

3.15 Must This action, activity, or procedural step is required.

3.16 Shall This action, activity, or procedural step is required.

3.17 Should This action, activity, or procedural step is suggested, but not required.

3.18 Service Request A form generated, at the time of sample receipt, which details pertinent information such as client name, address, contact, client and laboratory sample identifications, sampling and receipt dates and times, requested analyses, sample type, canister pressures (initial and final), and the service request number (unique number for each submitted job) and serves as an inter-laboratory "custody" form which accompanies all samples throughout the laboratory.

3.19 Air-Phase Petroleum Hydrocarbons (APH) These are defined as collective fractions of hydrocarbons compounds eluting from isopentane to n-dodecane, excluding target APH analytes. APH is comprised of C5-C8 aliphatic hydrocarbons, C9-C12 aliphatic hydrocarbons, and C9-C10 aromatic hydrocarbons.

3.20 APH Component Standard A mixture of the aliphatic and aromatic compounds listed in Table 4. The compounds comprising the APH Component Standard are used to define and establish the retention time windows for the collective aliphatic and aromatic hydrocarbon ranges of interest, and determine average chromatographic response factors that can in turn be used to calculate the collective concentration of hydrocarbons within these ranges. The APH target analytes are in a separate stock standard cylinder (also used for EPA Method TO-15) and are prepared as separate working standards in Summa canisters.

3.21 Laboratory Control Sample A humidified canister fortified with a gaseous-phase mixture of the APH Component Standard obtained from a different stock solution than the APH working/calibration standards.

4) Health and Safety Warnings

4.1 Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, Safety Data Sheet (SDS) location, and the laboratory waste management plan for the safe disposal of chemicals and samples.

4.2 Pollution Prevention and Waste Management

All waste disposals shall be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal.* In addition, canisters must be cleaned in accordance with the requirements detailed in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters.*

4.3 This procedure may include CHEMICAL, OPERATIONAL and/or EQUIPMENT hazards. Employees must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1: Standard and Sample Preparation	**Hazards**	**Preventative Measures**
Compounds, mixtures of compounds, standards, surrogates, and samples.	Exposure to potential health hazards through absorption through skin. Inhalation hazards.	Reduce exposure through the use of gloves and fume hoods. Safety glasses must be worn when working in the prep lab. Care should be taken when handling standard material in a neat or highly concentrated form. Personal protective clothing (safety glasses, gloves, and lab coat) are required when handling standard material in neat form. Consult Safety Data Sheets (SDS) for compounds being handled in this procedure, and be familiar with proper safety precautions.
Job Task #2: Working with Liquid Nitrogen	**Hazards**	**Preventative Measures**
Turning valves and handling tubing and fittings that have been in contact with the cryogen.	Can cause serious tissue damage (frostbite) with only a few seconds of contact.	Wear neoprene or leather gloves. Valves on cryogen dewars should be opened slowly so leaky fitting can be identified.
Job Task #3: Working with Pressurized Gases	**Hazards**	**Preventative Measures**
Using and moving compressed gas cylinders.	Gas leak, fire, and explosion. Personal injury due to falling during transport.	All cylinders must be secured in an upright position to a wall or immovable counter with a chain or a cylinder clamp when not in use. Keep safety caps on when cylinders are not in use. A handcart must be used when transporting cylinders. The cylinder must be secured to the handcart with a chain or belt. The regulator should never remain on small "D" size cylinders following use. Full cylinders must be kept separate from empty cylinders. Flammable gases (i.e. pressurized hydrogen) must be clearly labeled. Flammables and oxidizers must be separated by a ½-hour fire wall or by at least twenty feet.
Job Task #4: Glass Syringes	**Hazards**	**Preventative Measures**
Glass syringe use	Skin lacerations and punctures.	The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

5) Cautions

5.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

The instrument maintenance log must be kept current. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The entry in the log must include:

(a) the date of maintenance
(b) who did the maintenance
(c) description of the maintenance
(d) proof that the maintenance activity was successful

A notation of a successful tune and continuing calibration or initial calibration and the file number that accompanies the data will serve as proof that the maintenance is complete and the instrument is in working order.

The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, changing the pump oil, cleaning the source, or ordering a part. In addition, a notation should be made in the logbook stating that no samples were analyzed during the days that the instrument was down and no active maintenance was being conducted (i.e., where no other notation was made in the logbook for those days).

5.2 Concentrating Trap Routine maintenance includes periodic solvent cleaning of the Silcosteel lines in the valve oven if contamination is suspected. Also, periodic replacement of the multi-sorbent or partial replacement of the trap if analyte specific deterioration is detected is required. After repacking the trap it should be baked for a minimum of two hours (until a clean blank is generated), whereas a partial repacking requires baking the trap for a minimum of 20 minutes (or until a clean blank is generated).

5.3 GC System Column performance is monitored by observing both peak shapes and column bleed. Over time, the column will exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced (see Section 9.4). Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.4 Mass Spectrometer The Mass Selective Detector (MSD) ion source requires periodic cleaning to maintain proper performance. Symptoms of a dirty ion source include difficulty keeping the MSD in tune and fluctuating internal standard areas. The vacuum system should be serviced every six months, including changing the pump oil and checking the molecular sieve in the backstreaming trap.

5.5 Instrument Tuning The instrument is tuned with guidance from the procedure described in the Agilent Operations Manual, when necessary. The tune shall meet the tune criteria described in this document.

6) Interferences

6.1 Summa Canisters Canisters should be stored in a contaminant free location and should be capped tightly during shipment to prevent leakage and minimize any compromise of the sample. The pressure/vacuum is checked prior to shipment and upon receipt from the field. Any problems with the sample from the field are noted on the service request form and the Project Manager contacted.

Also, canisters must be cleaned and certified to be free from target analytes before being shipped to the field for sample collection. The procedure is described in detail in the *Standard Operating Procedure for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* (refer to this procedure as well as Section 12.7.1 for the acceptance criteria.).

6.2 Analytical System The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with buna-N rubber components must be avoided.

6.3 Glassware Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware results in discrete artifacts and/or elevated baselines in the detector profiles should be minimized. All glassware associated with this method must be scrupulously cleaned to avoid possible contamination. The cleaning shall be performed in accordance with the procedure outlined in the *SOP for Glassware Cleaning.* The use of high purity water, reagents, and solvents helps to minimize these problems.

6.4 Organic Compounds Certain organic compounds not associated with the release of petroleum products, including chlorinated solvents, ketones and ethers will be detected by this method and quantified within an aliphatic or aromatic hydrocarbon range. *When noted by the analyst, the identification and/or quantitation of such compounds must be disclosed on the laboratory report.* Non-APH compounds may be subtracted out of the hydrocarbon ranges before reporting results. When requested by the data user the identification of such non-APH compounds must be disclosed on the laboratory report or case narrative.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP may perform analysis, and interpretation of the results. The analyst must also ensure that a second analyst that is familiar with this analysis reviews the results and all applicable QC.

7.2 The supervisor/manager must ensure that method proficiency is documented initially and whenever significant changes in the instrument type, personnel, matrix or test

method are made.

7.3 The department supervisor/manager or designee shall perform final review and sign-off on the data.

7.4 All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy.* The training plan (Attachment 1) shall be used to document the training certification of new analysts.

8) Sample Collection, Handling, and Preservation

8.1 Air samples are collected in the field and delivered to the laboratory and should be collected in a specially prepared, leak-free, stainless steel pressure vessel (with valve) of desired volume (e.g., 6L). It is also acceptable to use Bottle Vacs (Entech Instruments, Simi Valley, CA) which are specially treated amber glass bottles fitted with a fused silica-coated valve (typically one liter volume). The use of Tedlar bags is considered a modification and is discouraged due to the inherent chemical artifacts which can interfere with the analysis.

8.2 Time-integrated samples require the use of a properly calibrated flow controller (refer to the Standard Operating Procedure for Flow Controllers and Critical Orifices). The flow controller must be calibrated prior to sample collection. Upon receipt at the laboratory, a post sampling calibration check must be performed on the flow controller. The relative percent difference (RPD) between the initial and post sampling calibration readings must be calculated. As long as the RPD is ≤20%, the calibration is considered to still be valid and thus the sample collection interval is also assumed to be valid. If the RPD is >20%, consideration must be given to whether resampling is necessary to achieve data quality objectives. If the sample is analyzed, a notation must be provided on the data reporting sheet and case narrative disclosing the RPD value.

8.3 There are no special preservation requirements for canisters. Canisters should be stored on the appropriate shelves until they are to be analyzed. The required holding time for samples in canisters for this method is 30 days.

9) Equipment and Supplies

9.1 Gas Chromatograph (GC) An instrument capable of temperature programming, with a column oven that may be cooled to sub-ambient temperature at the start of the gas chromatographic run to result in the resolution of the VOCs.

9.2 Autosampler

Teledyne-Tekmar AutoCan Autosampler:	14-ACAN-074
Concentrating Trap (cryogenic trap, built-in):	14-6938-020
Cryofocusing Module w/split valve:	14-6520-A00
GAST Vacuum Pump:	DOA-P104-AA

9.3 Mass Spectrometer (MS) A MS capable of scanning from 33 to 350 amu every second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

9.3.1 Ionization Gauge Controller

Granville-Phillips 330 Ionization Gauge Controller:	330001/2/3
Hewlett Packard Ionization Gauge Controller:	59864B

9.4 Analytical Column

Restek Rxi-1ms Fused Silica Capillary Column
60m x 0.25mm ID
1.0 micron film thickness

NOTE: Based upon data obtained from the MADEP VPH Round Robin testing programs, the choice of chromatographic column may have a significant impact on the apportionment and quantitation of aliphatic and aromatic compounds within the fractional ranges specified in this method. Substitution of the required column is not allowed, unless it can be demonstrated that the selected column has equivalent chromatographic properties and elution order for the aliphatic and aromatic compounds and ranges of interest.

To demonstrate equivalency of column chromatography, a mid-range calibration standard must be analyzed on both the required column and the proposed substitute column, with all other run and system parameters held constant. The concentrations of C5-C8 and C9-C12 aliphatic hydrocarbons, C9-C10 aromatic hydrocarbon ranges and target analytes must be determined for each column. The relative percent difference between the concentrations of each hydrocarbon range and target analyte, excluding naphthalene, obtained from each column must be ≤25. The RPD for naphthalene must be ≤40. The elution order of APH Components on the proposed substitute column must be equivalent to the elution order on the required column.

9.5 Data Systems IBM-compatible PC with Windows 95/98/NT/XP and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library or equivalent.

9.6 Canister Pressurization Station Vacuum/Pressure Gauge [0 to –30 in Hg; 0-90 psig]

9.7 Canister Sampling Devices VICI Condyne Model 300 Flow Controller

9.8 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent

10) Standards and Reagents

10.1 Reagents

10.1.1 UHP Grade Helium (99.999%)(GC carrier gas and preconcentrator purge/sweep gas)
10.1.2 Cryogen - Liquid nitrogen (used to cool preconcentrator traps)
10.1.3 UHP/Zero Grade Air
10.1.4 ASTM Type II Water or equivalent
10.1.5 High purity grade methanol

10.2 Standards

10.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Mixture Prepare a standard solution of p-Bromofluorobenzene (BFB-used as both a tune check and surrogate compound), bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene, 1,2-dichloroethane-d4(surrogate), and toluene-d8(surrogate) at 500ug/m^3 each in humidified zero air or nitrogen. This mixture may be purchased from an approved vendor in a high-pressure cylinder at the working concentration and Summa canisters filled directly from it for use on the sample preconcentrator. Otherwise, prepare this standard

according to the procedure outlined in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.

10.2.1.1 An intermediate standard can be prepared from neat compounds in a glass static dilution bottle (SDB). After the volume of the SDB is determined, calculate the mass of each compound to be spiked to achieve a final concentration of 5.0ug/mL. Then use the density of each neat compound to calculate the microliter amount to be spiked into the SDB. The SDB is then heated for a minimum of one hour at ~60˚C to completely volatilize all components.

Concentration of the intermediate standard prepared in a SDB is 5.0µg/mL. The amount required to achieve this concentration is determined through the use of the following equation.

$$A = \frac{(C)(V)}{D} \qquad \text{(Equation 1)}$$

Where:

A Amount of each compound required to achieve the desired concentration of the standard in the SDB (µL)
C Desired concentration of SDB (µg/mL)
V Actual volume of the SDB (mL)
D Density of the compound in question (µg/µL)

Example:

Calculate the amount of neat bromochloromethane needed to achieve the final concentration of 5.0µg/mL of that compound in the SDB.

V = 2010mL
D = 1934.4µg/µL
C = 5.0µg/mL

$$A = \frac{\left(5.0\frac{\mu g}{mL}\right)2010mL}{1934.4\frac{\mu g}{\mu L}} = 5.2\mu L$$

Table 1 – Tune, IS and Surrogate Compound Densities

Density (µg/µL)	Compound
1934.4	Bromochloromethane
1170.1	1,4-Difluorobenzene
1157	Chlorobenzene-d5
1307	1,2-Dichloroethane-d4
943	Toluene-d8
1593	BFB

10.2.1.2 The Working standard is prepared in a Summa canister by spiking an aliquot of the stock SDB standard (8.2.1.1) using a heated gastight syringe. Connect a cleaned, evacuated Summa canister to a source of

pure diluent gas (humidified zero air) using a teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum. Spike the SDB stock and following removal of syringe a small flow of diluent gas to flush the spike into the can. Pressurize the can to positive 83.3 psig with humid zero air, and allow the contents to equilibrate for approximately 24 hours before using.

Concentration of the working standard prepared in a Summa canister is 500ng/L. The final pressure of the canister is 83.3psig; therefore, the pressurized volume is 40L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 2)

Where:

PV Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PF = $\frac{P_{atm} + P_f}{P_{atm} + P_i}$

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister @ 1atm

P_{atm} Atmospheric Pressure = 14.7psig

Example:

$\frac{14.7 + 83.3}{14.7 + 0}(6L)$ = 40L

In order to prepare the canister with a concentration of 500ng/L, it must be determined how much of the intermediate standard is required. This is achieved through the use of the following equation.

$$A = \frac{(F)(V)}{(C)\left(1000\frac{ng}{\mu g}\right)}$$ (Equation 3)

Where:

F Desired concentration of working standard (ng/L)

V Pressurized Volume of Canister (L)

C Concentration of prepared SDB (µg/mL)

A Amount of standard (mL) of the SDB required to obtain the desired working standard concentration

Example:

$$A = \frac{500\frac{ng}{L}(40L)}{\left(5.0\frac{\mu g}{mL}\right)\left(1000\frac{ng}{\mu g}\right)} = 4mL$$

10.2.1.3 Currently the working standard is purchased in a cylinder at a certified concentration of 500ng/L (prepared by Liquid Technology Corporation). The working standard is filled directly into a summa canister to a pressure of 70 to 80 psig.

10.2.2 APH Component Standard (Stock Standard) Stock standards are purchased from an approved vendor as a mixture in a balance gas of nitrogen in high-pressure inert cylinders, and are available from several vendors. Each standard cylinder must be accompanied by a certificate of analysis stating the certified concentrations of each component. These concentrations must be used as the starting point when calculating the nanogram on-column amounts for the initial calibration points. See Table 5.

10.2.3 APH Working Standards Prepare gaseous-phase APH Working Standards at a minimum of two concentration levels in 6.0L Summa canisters pressurized with humidified zero air to 14.7psig. The contents should be allowed to equilibrate for approximately 24 hours prior to use.

Step 1: Concentration of the working standards prepared in Summa canisters should be 200ng/L and 20ng/L. The final pressure of the canister is 14.7psig; therefore, the pressurized volume is 12L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 6)

Where:

PV Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PF = $\frac{P_{atm}+P_f}{P_{atm}+P_i}$

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister @ 1atm

EXAMPLE:

$$\frac{14.7+14.7}{14.7+0}(6L) = 12L$$

Step 2: Use the Entech dynamic diluter to prepare the working standards in Summa canisters. The stock standard is typically at a concentration of 1000ng/L, so a 200ng/L can will be a 5X dilution, and the 20ng/L can will be a 50X dilution. Instructions for using the diluter and calculating flows can be

found in the instruction manual and in the TO-15 SOP (VOA-TO15).

10.2.4 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) For the second-source standard, use the TO-15 second source working standard. This standard contains all of the target analytes and at least one calibration compound from each hydrocarbon range.

Note 2: Any of the desired standard concentrations may change as long as the equations and the appropriate densities remain the same. In addition, the SDB volumes will change with each specific SDB utilized (indicated by the etched volumes on the specific SDB being utilized). The final pressures of the canisters may also change as long as the actual pressurized volumes are properly calculated in accordance with the corresponding equations detailed in this document. Use this section to calculate the alternate concentrations, pressurized volumes of the Summa canisters, etc., as needed.

10.3 Storage and Expiration Dates

- Static Dilution Bottle (SDB) standards (internal standard/surrogate) must be stored in an oven at a temperature of 60°C to ensure analyte vaporization. Every time a standard is prepared from the static dilution bottle (SDB), the concentration changes. To increase the useful lifetime of an SDB standard, remove volumes of 25mL or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, a SDB intermediate standard is stable for approximately *two months* as long as new working standards made from this standard continue to meet acceptance criteria. These bottles must be in the oven at 60°C for a minimum of one hour prior to use in preparing working standards.
- Stock Standard cylinders – These standards have an expiration date on the certificate of analysis (typically one year). Expired cylinders with sufficient volume remaining are sent back to the original vendor for recertification.
- APH Working Standards (excluding the ICV/LCS) prepared in canisters may be stored at laboratory conditions for *two months* in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

11) Method Calibration

11.1 Initial Calibration The APH Component Standards are used to calibrate the GC/MS system. Two distinct calibration operations are necessary:

Target APH Analytes: Relative Response Factors (RRFs) are calculated for the 9 Target APH Analytes (Table 4) and internal standards, based upon a correlation between the mass of analyte and area counts for the relevant quantitation ions. This allows for the individual identification and quantitation of these specific compounds. IT IS NOT NECESSARY TO DEVELOP RESPONSE FACTORS FOR ANY OTHER INDIVIDUAL APH COMPONENT STANDARD. However, an extended list of target analytes may be reported if needed since all the APH target analytes are included in the calibration for EPA Method TO-15 which is performed using the same GC and data acquisition parameters as the hydrocarbon range calibration.

Collective Aliphatic/Aromatic ranges: Relative Response Factors are calculated for C_5-C_8 Aliphatic Hydrocarbons and C_9-C_{12} Aliphatic Hydrocarbons based upon a correlation

between the TOTAL mass of aliphatic APH Component Standards eluting within the range of interest and the total ion area count. A Relative Response Factor is calculated for C_9-C_{10} Aromatic Hydrocarbons based upon a correlation between the TOTAL mass of aromatic APH Component Standards eluting within this range and the total area count of extracted ions 120 and 134. Specified APH Component Standards are designated "marker" compounds to define the beginning and end of the hydrocarbon ranges.

Primary and secondary extracted ions for all APH Component Standards and recommended internal standards are provided in Table 4. The recommended internal standards and associated Target APH Analyte and Hydrocarbon Ranges are provided in Table 3.

Table 3

Internal Standards and Associated Target APH Analytes and Hydrocarbon Ranges

Bromochloromethane (IS #1)	1,4-Difluorobenzene (IS #2)	Chlorobenzene-d5 (IS #3)
1,3-Butadiene Methyl tert-Butyl Ether	Benzene Toluene C_5-C_8 Aliphatics	Ethylbenzene m&p-Xylenes o-Xylene Naphthalene C_9-C_{12} Aliphatics C_9-C_{10} Aromatics

Table 4
Primary (Quantitation) & Secondary Ions for APH Component/Internal Standards

APH Component Standard	CAS Number	Mol Wt. (g/mol)	Target APH Analyte	Quantitation Ion	Secondary Ion(s)
Bromochloromethane (IS1)	74-97-5			130	49, 130
1,3-Butadiene	106-99-	54.09	✓	54	53, 39
Isopentane (Range Marker)	78-78-4			43	42, 41, 57
Methyl-tert-butyl ether	1634-	88.15	✓	73	57, 45
n-Hexane	110-54-			57	41, 43, 56
Cyclohexane	110-82-			56	84, 41
1,4-Difluorobenzene (IS2)	540-36-			114	88
2,3-Dimethylpentane	565593			56	43, 57, 41
Benzene	71-43-2	78.11	✓	78	52, 51
n-Heptane	142-82-			43	71, 57,
Toluene	108-88-	92.14	✓	91	92, 65
Chlorobenzene-d5 (IS3)	3114-			82	117
n-Octane	111-65-			43	85, 57, 71
Ethylbenzene	100-41-	106.17	✓	91	106
2,3-Dimethylheptane	3074-			43	84,85
m-Xylene	108-38-	106.17	✓	91	106, 105
p-Xylene	106-42-	106.17	✓	91	106, 105
n-Nonane (Range Marker)	111-84-			43	57, 85
o-Xylene (Range Marker)	95-47-6	106.17	✓	91	106, 105
Isopropylbenzene	98-82-8			105	120
1-Methyl-3-ethylbenzene	620-14-			105	120
1,3,5-Trimethylbenzene	108-67-			105	120
n-Decane	124-18-			57	43, 71, 85
Butylcyclohexane	1678-			83	55, 82
p-Isopropyltoluene	99-87-6			119	105, 134
1,2,3-Trimethylbenzene	526-73-			105	120
n-Undecane	1120-			57	43, 71, 85
n-Dodecane (Range Marker)	112-40-			57	43, 71, 85
Naphthalene (Range Marker)	91-20-3	128.17	✓	128	127, 102

Table 5
Standard Concentrations of APH Component Standards for Target APH Analytes and Hydrocarbon Ranges for Initial Calibration

Range	APH Component Standards used to Establish Range Response Factor	Calib. Level	Working Std conc (ng/L)	Injection Volume	Approximate Concentration
C_5-C_8 Aliphatic Hydrocarbons	Isopentane n-Hexane Cyclohexane 2,3-Dimethylpentane n-Heptane n-Octane	1	20	25mL	0.50ng
		2	20	50mL	1.0ng
		3	20	250mL	5.0ng
		4	200	125mL	25g
		5	200	250mL	50ng
		6	200	500mL	100ng
C_9-C_{12} Aliphatic Hydrocarbons	2,3-Dimethylheptane n-Nonane n-Decane Butylcyclohexane n-Undecane n-Dodecane	1	20	25mL	0.50ng
		2	20	50mL	1.0ng
		3	20	250mL	5.0ng
		4	200	125mL	25ng
		5	200	250mL	50ng
		6	200	500mL	100ng
C_9-C_{10} Aromatic Hydrocarbons	Isopropylbenzene 1-Methyl-3-ethylbenzene 1,3,5-Trimethylbenzene 1,2,3-Trimethylbenzene p-Isopropyltoluene	1	20	25mL	0.50ng
		2	20	50mL	1.0ng
		3	20	250mL	5.0ng
		4	200	125mL	25ng
		5	200	250mL	50ng
		6	200	500mL	100ng
Target APH Analytes	1,3-Butadiene Methyl tert-Butyl Ether Benzene Ethylbenzene m,p-Xylenes[b] o-Xylene Naphthalene	1	20	25mL	0.50ng
		2	20	50mL	1.0ng
		3	20	250mL	5.0ng
		4	200	125mL	25ng
		5	200	250mL	50ng
		6	200	500mL	100ng

[a] The actual concentrations shall depend on the certified analyte concentration from the applicable manufacturer's certificate of analysis.
[b] Xylene concentration is doubled.

11.1.1 Calibration Points Analyze a minimum of five levels of the calibration standard (analyze low to high) that span the monitoring range of interest of the samples. The range is typically 0.50ng to 100ng on column (m,p-Xylene is doubled). The dynamic range is dependent on the sensitivity of a particular instrument as well as the required reporting limit for a given project and may be adjusted accordingly. Refer to Table 5 for the approximate concentrations of the compounds of interest in the initial calibration. These concentrations may

change with the purchase and/or preparation of new standards; therefore, they should be verified.

The initial calibration is performed to determine instrument sensitivity and the linearity of the GC/MS response for the target compounds. One of the calibration points from the initial calibration curve must be at the same concentration as the continuing calibration verification standard. Also, one of the standards must be at or below the method reporting limit for the compounds of interest or the MRL must be adjusted accordingly.

11.1.2 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument or if the continuing calibration verification acceptance criteria have not been met as specified in Section 16.6.4.

11.1.3 Analytical Window If time remains in the 24-hour tune window after meeting the acceptance criteria for the initial calibration, samples may be analyzed according to the procedure described in this document. If time does not remain in the analytical window, a new sequence shall commence with the analysis of the instrument performance check compound (BFB) and the continuing calibration verification standard.

11.1.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 12.3. Use the standards prepared in accordance with Section 10 of this SOP. Attach the calibration standard and internal standard canisters to the designated inlets on the preconcentrator and open the canister valves. Analyzing different volume aliquots of the calibration standards produces differing concentrations. Internal standards must be added at the same volume for every standard, sample and QC sample.

Analyte responses (target ion areas) are tabulated and recorded using the Enviroquant program. Quantitation ions for the target compounds are shown in Table 4 and the primary ion should be used unless interferences are present, in which case the secondary ion may be used.

11.1.5 Initial Calibration Requirements

Initial calibration requirements are as follows:

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. Highest concentration, together with the lowest concentration, defines the calibration range.
3. Lowest concentration must be at or below the method reporting limit.
4. A blank should be analyzed prior to beginning the analysis of the calibration standards.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from low to high concentration.
8. All ICAL analyses must be completed within the 24-hour tune window.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at or below the MRL and may not be

dropped unless the MRL is changed to the concentration of the remaining lowest standard.

- Points at the high end may be dropped, but doing so lowers the calibration range.
- Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).
- Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.
- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 24 hours).
- Once the ICAL has been used to calculate and report sample results, it is not to be changed.

11.1.6 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not met and at least annually. The following procedure must be followed when updating an initial calibration method.

1. Open the most recent method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID must be the date files were analyzed.
3. Quantitate midpoint standard and check retention times and integrations. Update retention times if necessary using QEdit or Easy ID (Tools → Easy ID). Requant if any changes are made and verify all peaks are identified correctly. Print.
   a. While midpoint standard is loaded update reference spectra (Continuing Calibration → Update Reference Spectra).
   b. With midpoint standard loaded update qualifier ion ratios and retention times (Initial Calibration → Update Levels → Select Update Level and then select Retention Times (Replace) and Replace Qualifier Ion Relative Responses).
   c. If necessary adjust integration parameters prior to processing remaining ICAL points.
4. Quantitate remaining ICAL standards. Review each peak for retention time, integration, and print. Review low level standards for acceptable signal to noise ratios and high level standards for saturation.
5. All responses must be cleared from ICAL before updating (Initial Calibration → Clear All Calibration Responses).
6. Update responses for each standard level (Initial Calibration → Update Levels) or (Initial Calibration → Quick Levels Update). If Quick Levels Update is used do not requant datafiles.

7. Save method.
8. Check Response Factor Report and evaluate whether any points should be dropped following the criteria outlined in this SOP.
9. Save method if any changes are made.
10. Verify calibration files listed on Response Factor Report are correct.
11. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report.

11.1.7 Initial Calibration Review Analyst's calculation and assessment along with a peer review of all ICAL data and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples. In the case where samples are placed on the autosampler and allowed to run overnight, the sample results may only be reported if the ICAL is reviewed and found to be acceptable. The ICAL checklist in Attachment 2 must be used to document the review and approval process.

Analyte concentrations, which are not "real", not to be reported, or otherwise marked off the initial calibration, should be followed by a short explanation regarding the reason for the omission.

11.1.8 Initial Calibration File An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- BFB tune analysis report
- Blank analysis quantitation report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and %recovery report.

11.2 Initial Calibration Verification Standard Verify the initial calibration by analyzing an initial calibration verification standard (ICV). This standard shall be obtained or prepared from materials acquired from a different manufacturer or lot from that of the initial calibration and prepared according to Section 10.2.4. At a minimum, it must contain 1,3-butadiene, benzene, toluene, ethylbenzene, m-&p-xylene, o-xylene, and naphthalene, and at least one compound from each hydrocarbon range. Methyl tert-butyl ether may be included but may have wider recovery acceptance limits.

Inject 25ng or less (refer to the appropriate manufacturer's certificate of analysis for the actual secondary source standard concentrations) of the ICV standard depending on the dynamic range of a given instrument.

12) Sample Preparation/Analysis

12.1 Sample Preparation and Leak Check The initial pressure/vacuum is checked and the canister pressurized as needed upon receipt by the laboratory. Samples collected in canisters shall be pressurized with humidified zero grade air or Nitrogen. However, if the samples are to be analyzed in accordance with EPA Method 3C then the samples must be pressurized with UHP Helium. The client must be made aware of this in advance and given the option of either submitting two canisters for analysis or receiving a report with qualified results.

Canister Pressurization Samples must be pressurized (to approximately 3.5psig) prior to analysis with humidified zero air (refer to exception stated above). This may be

accomplished by connecting the sample canister to a source of pure diluent gas (zero air) using a teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum and injecting 100uL of water into the can through the septum and allowed to vaporize for approximately 10 minutes. Alternatively, pressurize at a fill station by bubbling the diluent gas through a zero air bubbler. Both of these procedures shall utilize ASTM Type II water or equivalent. Additional information may be found in the *Standard Operating Procedure for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters*. Initial and final pressures shall be recorded and the dilution factor created by filling the sample canister is calculated using Equation 25 in Section 15.3.4.

Leak Check Connect the canister(s) to the autosampler. Place a ¼" stainless steel nut and ferrule on the inlet line facing the canister. Push the inlet line into the orifice of the canister and hold in place while tightening the fitting finger tight. Turn the stainless steel nut ¼ turn more with a wrench. The canister valves should be closed at this point. For Bottle Vacs, connect the female Micro-QT fitting to the autosampler. A leak check must be performed before connecting the sample bottles since the valve is open as soon as the bottle is connected.

Leak Checks – Leak check all canister inlet connections. Analysis may not begin until the leak check has passed for each canister being tested. If a leak is detected, it should be confirmed by placing on a different location. In addition, the valve threads should be inspected for defects which may prevent a good seal with the AutoCAN. Once a canister has "failed" the leak check it must be tagged, an NCAR initiated, and the PM notified. Regardless of what the client or PM specifies as the fate of the sample, the canister must be put on maintenance hold to complete a full 24-hour leak check. A yellow sheet is to be completed in addition to, but not in lieu of an NCAR. This is a fixed QA procedure with no allowance for deviation.

12.2 Analytical Sequence

12.2.1 Analytical Sequence For this internal standard calibration method analysis, a CCV standard is to be analyzed every 24 hours. That is, the last analysis in the sequence must be started within 24 hours from the time of the initiation of the sequence. The initiation is considered to be the injection of the BFB tune standard.

The analytical sequence must be completed for the analysis of ≤20 field samples. A method blank (MB) shall be run to monitor for laboratory introduced contamination. There must be at a minimum a laboratory duplicate (LD) analyzed in each batch to access batch precision. A laboratory control sample (LCS) shall be analyzed at a rate of at least one per batch of twenty or fewer samples. The concentration of the LCS (ICV standard) should be at the lower end of the calibration curve as an indication that the system allows for good recovery at those concentrations. The following is the analytical sequence guideline for this method.

Analytical Sequence Guideline

With Calibration	Tune Check[1]
	Calibration Standards (5 Standards Minimum)
	ICV Standard[2] (Acts as the ICV and LCS)
	QC Canister Checks[6]
	MB[7]
	Sample(s)
	Laboratory Duplicate[4]

With Continuing	Calibration Tune Check[1]
	CCV Standard[5]
	QC Canister Checks[6]
	MB[7]
	LCS[3]
	Sample(s)
	Laboratory Duplicate[4]

[1] The introduction of the tune check standard is the start of the 24 hour analysis window. The instrument performance check solution must be analyzed initially and once per 24 hour time period of operation.
[2] In this scenario, the ICV may also be evaluated as the LCS.
[3] An LCS shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard.
[4] A laboratory duplicate must be analyzed at a rate of 1 per 20 or fewer samples. The duplicate must be reported even if it is a batch duplicate.
[5] A CCV must be analyzed at the beginning of every analytical sequence.
[6] Any number of QC check canisters may be analyzed in the sequence to determine a canister cleaning batch or batches acceptability.
[7] Any of the QC Check Canisters may serve as the method blank as long as the minimum requirements detailed in this document are met. A method blank shall be analyzed at a rate of 1 in 20 or fewer samples.

12.3 Conditions

12.3.1 Sample Collection Conditions The suggested settings and system parameters are as follows:

Adsorbent Trap

Set Point:	40˚
Sample Volume:	25ml to 1,000ml
Dry Purge:	300mL
Sampling Rate:	100ml/min or 40ml/min
Desorb Temp.:	210˚C
Desorb Flow Rate:	8-10mL/min He
Desorb Time:	3.0 minutes

Refocusing Trap

Temperature:	-175˚C
Injection Temp.:	150˚C
Injection Time:	1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature:	10°C above desorb temperature
Initial Bakeout:	2 hours or until clean blank is obtained
After each run:	10 minutes

12.3.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

Item	Condition
Carrier Gas	Helium
Flow Rate	1.0-1.6mL/minute
Temperature Program	Initial Temperature: 10˚C Initial Hold Temperature: 1 minute Ramp Rate: 5˚C/min to 50˚C 2nd Ramp: 10˚C/min to 100˚C 3rd Ramp: 20˚C/min to 240˚C for 4 min hold
Detector B (MSD *Interface)*:	260˚C
Electron Energy	70 Volts (nominal)
Mass Range	33 to 280 amu (SCAN mode)
Scan Time	To give at least 10 scans per peak, not to exceed 1 second per scan.

12.4 Retention Time Windows The laboratory should calculate retention time windows initially and whenever a new GC column is installed. The laboratory must retain these data.

Before establishing retention time windows, ensure that the GC/MS system is operating within optimum conditions. Analyze an APH Calibration Standard on three separate occasions throughout the course of a 72-hr period. Serial analyses over less than a 72-hr period may result in retention time windows that are too restrictive.

Calculate the standard deviation of the three absolute retention times for each Target APH Analyte, range "marker" compound, internal standard, and MS tuning standard.

The retention time window is defined as plus or minus three times the standard deviation of the absolute retention times for each analyte of interest. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

In those cases where the standard deviation for a particular standard approaches zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop an operational retention time window.

Table 2
APH Range "Marker" Compounds and Range Retention Time Windows

Hydrocarbon Range	Beginning Marker	Ending Marker
C_5-C_8 Aliphatic	0.1 min. before isopentane	0.01 min. before n-nonane
C_9-C_{12} Aliphatic Hydrocarbons	0.01 min. before n-nonane	0.1 min. after naphthalene*
C_9-C_{10} Aromatic Hydrocarbons	0.1 min. after o-xylene	0.1 min before naphthalene

*The method specifies using n-dodecane as this marker, but in practice naphthalene elutes after n-dodecane so the laboratory must use naphthalene as the marker.

The relative retention time (RRT) and RRT window for each Target APH Analyte, internal

standard, and hydrocarbon range "marker" compound must be verified on a daily basis. The RRT for each analyte of interest shall be established as the midpoint of the window. The retention time window equals the midpoint ± three times the standard deviation (Equation 9).

12.5 Instrument Performance Check Since the BFB tuning compound is included in the internal standard canister and a autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

The following is the procedure to follow when performing the instrument performance check.

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.

All subsequent standards, samples and QC samples associated with a BFB analysis must use identical instrument conditions. Refer to Section 16.6.1 (Table 7) for the acceptance criteria and required corrective action.

12.6 Continuing Calibration Verification Standard Verify the calibration each working day, where necessary (e.g., an ICAL was not analyzed or the 24-hour tune window has closed) by analyzing a continuing calibration verification (CCV) standard from the initial calibration standard canister. The concentration of the calibration verification should be varied within the established calibration range.

12.7 Canister Quality Control Check and Method Blank A Quality Control (QC) check canister may also serve as a method blank (see note 1 below) as long as the analyte concentration requirements stated in the canister quality control check section (Section 12.7.1) and the other requirements (refer to Section 16.6.7 for internal standard requirements) are met. If a QC canister fails with respect to the analyte concentration criterion, it may still be used as a method blank as long as the method blank criteria stated in 12.7.2 are met. If a QC canister still fails, another QC canister or a new canister must be prepared and analyzed (per Section 12.7.2) in order to verify that no system contamination exists.

Note 1: The use of a QC canister as a method blank is considered acceptable since a canister that has been sent into the field, returned and cleaned more closely resembles the manner in which client samples are handled.

12.7.1 Canister Quality Control Check The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *Standard Operating Procedure for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included.

The canister to be checked, shall be pressurized with humidified zero grade air prior to analysis. Analyze an aliquot of one liter along with the same volume of internal standard as standards and samples. The unique laboratory barcode given to a canister shall be the information included in the sample analysis

identification, which is for tracking purposes. A canister is considered "clean" if the analysis shows <0.2ppbv of any target analyte or hydrocarbon range (refer to Note 1).

12.7.2 Method Blank In order for a method blank to be considered acceptable all target analytes must be less than the method reporting limit and fulfill the additional requirement in Section 16.6.5. If the QC canister(s) fail the corresponding criteria then the following must be performed.

- Prepare a canister that has not left the building by pressurizing with humidified zero air.
- Analyze an aliquot of the blank (1 liter) with internal standard
- Be consistent with the volume of internal standards introduced for each analysis.

Additionally, analyze a method blank whenever a high concentration sample is encountered and carryover is suspected.

The analyst should cross out those concentrations that are not real and initial and date the quantitation report for those QC Check canisters and method blanks that meet the acceptance criteria included in this section.

12.8 Laboratory Control Sample The laboratory control sample is an injection of the initial calibration verification standard. Inject the LCS (ICV) at concentrations at or below the midpoint of the calibration curve. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

12.9 Sample Analysis Prior to analysis, all sample containers should be at temperature equilibrium with the laboratory.

- Attach sample canisters Tekmar AUTOCan using a 9/16" wrench. Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments).
- Before opening the valve, check for leaking fittings by running the leak check program in the Teklink software. Quick connect fittings must be leak checked before connecting the sample container.
- If system is leak tight, open the canister valves and start the automated preconcentration procedure. Make sure the Chemstation data acquisition software has been readied.
- Maintain the trap at an elevated temperature until the beginning of the next analysis.
- Introduce the same volume of internal standards as used for the standards and QC samples.

Note 1: The secondary ion quantitation is only allowed if there is sample matrix interference with the primary ion. If the secondary ion quantitation is performed, document the reasons in the instrument run logbook and/or on the quantitation report (initial and date any notation).

Note 2: Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fitting higher than 80ºC.

12.9.1 Qualitative Identifications The Target APH Analytes must be identified by an analyst competent in the interpretation of chromatograms and mass spectra.

Two criteria must be satisfied to verify the identification: (1) elution of the component in the sample at the same GC relative retention time (RRT) as the component in the standard, and (2) agreement of the sample component and standard component mass spectra.

If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned using extracted ion current profiles for the ion unique to the component of interest.

For comparison of the standard and sample component mass spectra, mass spectra of standards obtained on the GC/MS under the same instrument conditions are required. Once obtained, these standard spectra may be used for identification and reference purposes. The requirements for qualitative verification by comparison of mass spectra are as follows:

All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

The relative intensities of ions specified must agree within ±20% between the standard and sample spectra.

Ions greater than 10% in the sample spectrum must be considered and accounted for by the analyst making the comparison.

The primary and secondary ions for all APH Component Standards are provided in Table 4.

12.9.2 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot should be analyzed. The smallest volume used shall not be less than that used for the initial calibration (see Table 5). The dynamic range of volume aliquots for the automatic cryogenic concentrator is 15ml to 1L. If a volume smaller than 15ml is to be analyzed, a dilution should be made in a Tedlar bag, or the sample directly injected using a gastight syringe. Guidance in performing dilutions and exceptions to this requirement are given below.

- Use results of the original analysis to determine the approximate dilution factor required and get the largest analyte peak within the initial calibration range.
- The dilution factor chosen should keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument.
- All dilution factors (Equation 25) must be documented and included in the final report.

Note: Refer to Section 18.7.3 for requirements on reporting results outside of the initial calibration range.

12.10 Manual Integration The integration for each peak shall be checked to ensure that it has been integrated properly. Assuming an incorrect automatic integration the analyst shall conduct the manual integration in accordance with the *SOP for Manual Integration Policy* including all documentation and reviews associated with the process. The review shall include the analyst and reviewer initialing and dating the manual integration as an indication of acceptability and approval.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Data System Setup

For the Tekmar AutoCan, fill in the sequence log of the Teklink program with the appropriate information. Refer to the Section 12.3.1 for the operating parameters.

For the HP Chemstation, load the appropriate acquisition method for the GC/MS in the top window of the Chemstation program. Suggested GC/MS operating parameters are given in Section 12.3.2.

14.2 Storing Electronic Data The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. Therefore, files will be named with an eight-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files are saved in a unique sub-directory on the server.

14.3 Sufficient raw data records must be retained of the analysis, instrument calibrations and method detection limit studies including: analysis/calibration date and time, test method, instrument, sample identification, analyte identification, analyst's initials, concentrations and responses, as well as standards used for the analysis and calibrations, all manual calculations including sample dilutions and manual integrations to permit reconstruction of analyses. Information entered and reported on the quantitation report and instrument run log must be complete and accurate. Retain all daily QC per sequence on file for future reference including tune checks, opening standards, method blanks, laboratory control samples, laboratory duplicates, and initial calibrations and initial calibration verifications. Additionally, all passing QC Canister checks must also be retained on file.

Note: All data records must explicitly connect data to the initial instrument calibration. This includes all samples, continuing calibrations and QC samples.

14.4 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings and balance gas if pressurized with helium), standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 Initial Calibration Calculations

15.1.1 Target APH Analytes Quantitation of the target analytes is done using the same data analysis method used for EPA TO-15 since all the APH target analytes are part of the laboratory's TO-15 analyte list. Tabulate the area response of the characteristic ions against the mass of each Target APH Analyte and internal standard and calculate relative response factors (RRFs) for each compound

using Equation 12. Perform this calculation for each Target APH Analyte.

Equation 12: Relative Response Factor for Target APH Analytes

$$RRF = [(A_{EC})*(C_I)] / [(A_{EI})*(C_c)]$$

where:

RRF = relative response factor
A_{EC} = area count of the extracted ion for the analyte of interest
C_I = mass of internal standard (ng)
A_{EI} = area count of the extracted ion for the associated internal standard
C_C = mass of analyte of interest (ng)

15.1.2 Hydrocarbon Ranges Calculate a response factor for the C_5-C_8 Aliphatic Hydrocarbon range using the following steps.

Using total ion integration, sum the individual peak areas of the six (6) APH Component Standards that are used to establish an average range response factor for C_5-C_8 Aliphatic Hydrocarbons, as designated in Table 5. Do not include the peak areas of internal/tuning standards.

Using the total area generated, calculate the Range RRF using Equation 13.

Equation 13: Relative Response Factor for C_5-C_8 Aliphatic Hydrocarbons

$$\text{Range } RRF = [(A_T)*(C_I)] / [(A_{EI2})*(C_T)]$$

Where:

Range RRF = relative response factor for the hydrocarbon range
A_T = total ion area count of the six aliphatic APH Component Standards which elute within this range (see Table 5)
C_I = mass of internal standard #2, ng (1,4-Difluorobenzene)
A_{EI2} = area count of the extracted ion for internal standard #2
C_T = summation of the masses of the six aliphatic APH Component Standards (ng) which elute within this range (see Table 5)

15.1.2.1 Calculate a response factor for the C_9-C_{12} Aliphatic Hydrocarbon range using the following steps.

Using total ion integration, sum the individual peak areas of the six (6) APH Component Standards that are used to establish an average range response factor for C_9-C_{12} Aliphatic Hydrocarbons, as designated in Table 5. Do not include the peak areas of internal/tuning standards.

Using the total area generated, calculate the Range RRF using Equation 14.

Equation 14: Relative Response Factor for C_9-C_{12} Aliphatic Hydrocarbons

$$\text{Range } RRF = [(A_T)*(C_I)] / [(A_{EI3})*(C_T)]$$

where:

Range RRF = relative response factor for the hydrocarbon range
A_T = total ion area count of the six aliphatic APH Component Standards which elute within this range (see Table 5)
C_I = mass of internal standard #3, ng (Chlorobenzene d5)
A_{EI3} = area count of the extracted ion for internal standard #3
C_T = summation of the masses of the six aliphatic APH Component Standards (ng) which elute within this range.

15.1.2.2 Calculate a response factor for the C_9-C_{10} Aromatic Hydrocarbon range using the following steps.

Using extracted ion 120, sum the individual peak areas of the five (5) APH Component Standards that are used to establish an average range response factor for C_9-C_{10} Aromatic Hydrocarbons (only four of the compounds will contribute area from m/z 120), as designated in Table 5. Do not include the peak areas of internal/tuning standards.

Using extracted ion 134, sum the peak areas of the five (5) APH Component Standards that are used to establish an average range response factor for C_9-C_{10} Aromatic Hydrocarbons (only one compound will contribute area from m/z 134), as designated in Table 5. Do not include the peak areas of internal/tuning standards.

Sum the area counts from each extracted ion.

Using the area count generated, calculate the RRF using Equation 15.

Equation 15: Relative Response Factor for C_9-C_{10} Aromatic Hydrocarbons

$$\text{Range } RRF = [(A_T)*(C_I)] / [(A_{EI3})*(C_T)]$$

where:

Range RRF = relative response factor for the hydrocarbon range
A_T = summation of area counts using extracted ions 120 and 134
C_I = mass of internal standard #3, ng (Chlorobenzene d5)
A_{EI3} = area count of the extracted ion for internal standard #3
C_T = summation of the masses of the five aromatic APH Component Standards (ng) which elute within this range (see Table 5)

Calculate the average response factor for each of the Target APH Analytes and each hydrocarbon range.

Calculate the percent relative standard deviation (%RSD) of the response factors over the working range of the curve for each of the Target APH Analytes and each hydrocarbon range using Equation 16.

Equation 16: Percent Relative Standard Deviation

This equation is also used for initial demonstration of capabilities, method detection limits studies, and method reporting limit verifications.

$$\%RSD = [(SD_{n-1}) / (AVG_X)]*100]$$

where:

%RSD = percent relative standard deviation
SD_{n-1} = standard deviation (n-1 degrees of freedom)
AVG_x = average response factor from the initial calibration curve

15.2 Sample Calculations

15.2.1 Individual Target APH Analytes The average response factor from the initial calibration is used to calculate the amount of analyte detected in the sample. Equation 17 is used to calculate the mass of sample analyte in ng. Equation 18 is used to convert ng to µg/m³. Equation 19 is used to convert of µg/m³ to ppbV.

Equation 17: Calculation of Analysis Results in ng

$$ng = [(A_x)*(C_{IS})] / [(A_{IS})*(RRF_{avg})]$$

where:

A_x = area of quantitation ion for the Target APH Analyte (see Table 4)
C_{IS} = mass of the internal standard
A_{IS} = area of quantitation ion for the associated internal std (see Table 4)
RRF_{avg} = average response factor for the specific compound to be measured**

Equation 18: Conversion of ng to µg/m³

$$\mu g / m3 = (ng / VA) * DF$$

where:

V_A = volume of sample analyzed (liters)
DF = dilution factor (Equation 25); if no dilution was made, the dilution factor =1

Equation 19: Conversion of µg/m³ to ppbV

$$ppbV = (\mu g / m3) * 24.46 / MW$$

where:

MW = molecular weight of the compound of interest, g/mol (see Table 4 for a list of the molecular weights of the Target APH Analytes)

15.2.2 Hydrocarbon Ranges The average range response factor from the initial calibration is used to calculate the mass (ng) of range hydrocarbons in samples. Collective peak area integration for the hydrocarbon ranges must be from baseline to baseline (i.e., must include the unresolved complex mixture).

15.2.3 The contribution of compounds not meeting the definition of aromatic or aliphatic hydrocarbons may be omitted from the collective hydrocarbon range calculations at the discretion of the laboratory and the data user. Only peaks with a peak height greater than half of the nearest internal standard need to be evaluated for exclusion. The guidance for making this decision includes the following:

- If the non-APH compound co-elutes with an aliphatic petroleum hydrocarbon, the area may not be subtracted from the aliphatic range.

- In complex sample matrices (i.e. many co-eluting peaks, complex petroleum patterns), this type of data adjustment may not be possible.
- Spectral identification of the excluded peak must be evaluated by a qualified mass spectrometrist. The analyst should consider the quality of the spectral library match, presence and relative intensity of major ions, and potential interferences in making a professional judgment on exclusion.

C_5-C_8 Aliphatic Hydrocarbons

- Using total ion integration, sum all peaks in the appropriate retention time window as specified in Sections 12.4 and Table 2.
- From this sum, subtract the total ion area counts of all internal standards and surrogates which elute in this range (all three of the recommended internal standards and two of the surrogates elute in this range). Also subtract the total ion area counts of all non-APH compounds that are not to be included in the final result.
- Calculate a preliminary mass amount in ng using Equation 20.

  Equation 20: Calculation of Preliminary Sample Analysis Results (ng)

  $$ng = [(A_x)*(C_{IS})]/[(A_{IS})*(RRF_{avg})]$$

  where:

  A_x = total ion area count of all peaks eluting within C5-C8 Aliphatic Hydrocarbon range window
  C_{IS} = mass of the internal standard, ng
  A_{IS} = area of quantitation ion for internal standard #2 (1,4-Difluorobenzene)
  RRF_{avg} = average range response factor for the C5-C8 Aliphatic Hydrocarbon range

- From the preliminary amount (ng), calculate an adjusted mass amount of range hydrocarbons by subtracting the masses of Target APH Analytes which elute in this range (MtBE, benzene, toluene, ethylbenzene, m-xylene, p-xylene, and o-xylene,).
- Convert the adjusted ng value to µg/m³ using Equation 21.

  Equation 21: Conversion of ng to µg/m³

  $$\mu g/m^3 = (C_{ng}/V_A)*DF$$

  where:

  C_{ng} = adjusted total mass of range hydrocarbons in ng
  V_A = volume of sample analyzed (liters)
  DF = dilution factor (Equation 25); if no dilution was made, the dilution factor = 1.

C_9-C_{10} Aromatic Hydrocarbons

- Using extracted ion 120, sum all peaks in the appropriate retention time window as specified in Section 12.4 and Table 2.
- Using extracted ion 134, sum all peaks in the appropriate retention time window as determined in Section 12.4 and Table 2.
- Sum the areas of ions 120 and 134.

- Subtract the extracted ion area (mass 120 and 134) of any non-APH compounds that are not to be included in the final result.
- Calculate an amount in ng using Equation 20, using the summed areas of ions 120 and 134.
- Convert the ng value to µg/m³ using Equation 21.

C_9-C_{12} Aliphatic Hydrocarbons

- Using total ion integration, sum all peaks in the appropriate retention time window as specified in Section 12.4 and Table 2.
- From this sum, subtract the total ion area counts of the 4-bromofluorobenzene (Surrogate #3) peak.
- Subtract the total ion area counts of all non-APH compounds that are not to be included in the final result.
- Calculate a preliminary mass amount in ng using Equation 20.
- From the preliminary amount (ng), calculate an adjusted mass amount of range hydrocarbons by subtracting the masses of Target APH Analytes which elute in this range (naphthalene), and by subtracting out the mass amount of C_9-C_{10} Aromatic Hydrocarbons.
- Convert the ng value to µg/m³ using Equation 21.

15.3 Additional Calculations

15.3.1 Relative Percent Difference This equation is used for laboratory duplicates and post calibration check of flow controllers when they are received back by the laboratory following sampling.

Equation 22: Relative Percent Difference

$$\frac{x_1 - x_2}{\bar{x}} \ (100)$$

where:

x_1 First measurement value
x_2 Second measurement value
$\bar{x}$ Average of the two values

15.3.2 Percent Difference This equation is used for the continuing calibration verification standards.

Equation 23: Percent Difference

$$\%D = [(RF_C) - (RF_I)] / [(RF_I)] * 100$$

where:

%D = percent difference
RF_C = response factor from the continuing calibration verification standard
RF_I = average response factor from the initial calibration curve

15.3.3 Percent Recovery This equation is used for the initial calibration verification standard, laboratory control sample, initial demonstration of capability, method detection limit study, and method reporting limit verifications.

Calculate the percent recovery (%R) of the Target APH Analyte or hydrocarbon range using Equation 24.

Equation 24: Percent Recovery

$$\%R = [(C_{found}) / (C_{true})]*100$$

%R = percent recovery
C_{found} = mass of the analyte or hydrocarbon range detected in the standard (ng)
C_{true} = true mass of the analyte or hydrocarbon range in the standard (ng)

15.3.4 Dilution Factors

Equation 25: Dilution Factor for Pressurization of Subatmospheric Samples:

$$PDF = \frac{P_{atm} + P_f}{P_{atm} + P_i}$$

where:

P_{atm} is the ambient atmospheric pressure, 14.7 psi at sea level.
P_f is the final sample canister pressure, in psig.
P_i is the initial sample canister pressure, in psig. This will most often be a negative value (sub-ambient initial pressure.)

15.3.5 Relative Retention Time

Equation 26: Relative Retention Time (RRT)

$$RRT = \frac{RT_C}{RT_{is}}$$

where:
RT_C Retention time of the target compound, seconds.
RT_{is} Retention time of the internal standard, seconds.

15.4 Data Review The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated by analytical sequence following the data review checklist in Attachment 3. The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second analyst. The data review checklist is used to document the reviews and once it has been completed, initialed and dated it must be filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file organized by instrument and date. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.8.

15.5 Reporting The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and all information required by the reference method and the laboratory quality control program.

In addition to sample results, the APH data report must include the following items:

- Method Blank results
- LCS results

- Sample duplicate results
- Internal standard results (areas) for all field samples and QC samples

15.5.1 Analysis Observations / Case Narrative Summary Form This form, which is included in the *SOP for Sample Analysis, Storage and Tracking*, must be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. This form is necessary as a means for documentation and will be reviewed along with other information when compiling the final report and case narrative.

Any sample flow controller that does not meet the post calibration check criteria (refer to Section 8.2) must be noted on this form so that it may be reported to the client.

All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

15.5.2 Significant Modifications "Significant Modifications" to the APH Method shall include, but are not limited to, any of the following and must be reported accordingly, if they occur.

(1) The use of sample collection devices other than evacuated passivated stainless steel canisters or glass Bottle Vacs (i.e., Tedlar bags).
(2) The use of alternative detectors other than GC/MS to quantify Target APH Analytes and/or hydrocarbon range concentrations.
(3) The use of extracted ions other than 120 and 134 to quantify C9-C10 aromatic hydrocarbons.
(4) The failure to provide all of the data and information required in the report form presented in Appendix 3.

Data produced using an analytical method incorporating any of the "Significant Modifications" described above may *not* be reported as APH data. APH range concentrations are method-defined parameters and as such may only be reported as APH data when produced using the method without "Significant Modifications."

Helium Pressurization – If a canister is pressurized with helium, a correction factor is applied to sample volumes extracted from the canister via auto sampler. This is due to the difference in thermal properties between helium and air. A correction factor worksheet has been generated to determine the exact volume taken from a canister and may be found at J:\\A-GCMS\Helium Pressurization (save the job as P1_____h.xls). Print the sheet and include with the data. Refer to the instruction page in the template for all of the instructions and calculations including backfilled canisters.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 This section of the standard operating procedure contains technical acceptance criteria. To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.2 Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV. Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action,* where appropriate.

16.3 Analytical Sequence Refer to Section 12.2.1 for the analytical sequence requirements. All analytical sequences and data must be recorded in an instrument run logbook.

16.4 Minimum Instrument QC (Additional) The following are additional requirements or are reiterated from previous sections.

- Internal standards used must be adequately resolved from individual compounds in the APH Component Standard.
- Retention time windows and relative retention times must be established for each target APH analyte, range "marker" compound, and internal standard initially and each time a new GC column is installed, and must be verified and/or adjusted on a daily basis (see Section 16.6.4).

16.5 Initial and Periodic Method QC Demonstrations The following procedures must be conducted as an initial demonstration of laboratory capability (IDLC). Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method or operational problems.

16.5.1 Accuracy and Precision To demonstrate initial laboratory capability, analyze a minimum of four replicate samples obtained from a humidified canister fortified with each Target APH Analyte.

- Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte and hydrocarbon range, and the replicate precision (as %RSD) of the measurements for each analyte.
- For each analyte and hydrocarbon range, the mean accuracy, expressed as a percentage of the true value, must be between 70% and 130%, and the %RSD must be less than or equal to 25. The IDLC must meet these conditions for analysis to proceed.
- If desired, the accuracy and precision evaluation may be combined with the MDL evaluation specified in Sections 16.5.2 and 16.5.3.

16.5.2 Method Detection Limits for Target APH Analytes Although the method does not require that MDL studies be performed, the APH target compounds are a subset of the EPA TO-15 analysis for which laboratory performs annual MDL determinations as follows. Analyze a minimum of seven replicate samples obtained from a canister fortified with all Target APH Analytes of interest at 3 to 5 times the calculated or estimated Instrument Detection Limits (IDLs) or at the low level initial calibration standard concentration. Analyze each replicate according to the procedures described in this document. Calculate the Method Detection Limit (MDL) of each analyte using Equations 9 and 10 and Table 6 below.

Equation 9: Standard Deviation

$$SD = \sqrt{\sum_{i=1}^{N} \frac{\left(C_i - \overline{C}\right)^2}{N-1}}$$

where:

C_i are the individual concentrations from each MDL replicate analysis

$\overline{C}$ Average (or Mean) concentration of all MDL replicate analyses

N total number of MDL replicate analyses

Equation 10: Method Detection Limit

$$MDL = (t) \times (SD)$$

where:

t = student t value at the 99% confidence level.
SD = standard deviation of the replicate analysis.

Table 6
Student t Values

Number of replicates	t value
7	3.143
8	2.998
9	2.896
10	2.821

16.5.3 Method Detection Limits for Hydrocarbon Ranges The method does not require that MDL studies be performed. However, the laboratory may choose to perform them in anticipation of client requests. Analyze a minimum of seven replicate samples obtained from a humidified canister fortified with all of the APH range calibration compounds at 3 to 5 times the calculated or estimated Instrument Detection Limit (IDL) or at the low level initial calibration standard concentration. Analyze each replicate according to the procedures described in this document. Calculate the Method Detection Limit (MDL) of each range using Equations 9 and 10 and Table 6.

16.5.4 Method Reporting Limits

16.5.4.1 Target APH Analytes The method reporting limit for each target APH analyte must be at or above the low level calibration standard and should be verified on an annual basis by analyzing at least 4 replicate samples from a canister spiked at the reporting limit, where the precision is demonstrated to be equal to or less than 25% RSD, and the mean accuracy is demonstrated to be between 70%-130% of the spiked value.

16.5.4.2 Collective Hydrocarbon Ranges The method recommends that the MRL for each hydrocarbon range be based upon the concentration of the lowest range calibration standard for the components that make up this range. The minimum MRL for each range is equal to the sum of the mass amounts of all the individual components in the lowest calibration standard point that are used for creating that range's RRF. In practice, this leads to MRLs that are so low that chromatographic

baseline noise often yields a false positive result. The laboratory will set the MRLs at or above this level so long as it meets the data quality objectives of the data user.

16.6 Ongoing Method QC Demonstrations

16.6.1 Instrument Performance Check

Acceptance Criteria – The GC/MS system must meet the mass spectra ion abundance criteria listed in Table 7. The appropriate corrective action is described below. Results of the BFB tune check as well as any actual tuning must be recorded and a copy of the tune report maintained on file.

Table 7 BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	8.0 – 40.0 percent of the base peak
75	30.0 – 66.0 percent of the base peak
95	base peak, 100 percent relative abundance
96	5.0 – 9.0 percent of the base peak
173	less than 2.0 percent of mass 174
174	50.0 to 120.0 percent of the base peak
175	4.0 – 9.0 percent of mass 174
176	greater than 93.0 percent but less than 101.0 percent of mass 174
177	5.0 – 9.0 percent of mass 176

Corrective Action – Re-analyze the BFB compound or perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria.

16.6.2 Initial Instrument Calibration

Acceptance Criteria

- Refer to Section 11.1.5 for the initial calibration procedure requirements (i.e., number of points, dropping points, etc.)
- The calculated percent relative standard deviation (%RSD, linear or quadratic regression is not allowed) for the relative response factors (RRF) for each compound in the calibration standard must be ≤30% with *Naphthalene* up to ≤40%.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable initial calibration verification (ICV) (refer to Section 16.6.3).

Corrective Action – Follow the initial calibration guidelines detailed in this document for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards. If the criteria are not met it may be necessary to perform maintenance, if this is the case then all calibration points must be re-analyzed.

16.6.3 Initial Calibration Verification Standard (ICV) / Laboratory Control Sample (LCS)

Acceptance Criteria – The spike recovery (%R) must be between 70%-130%.

Corrective Action – If the technical acceptance criteria are not met, reanalyze and if it still fails prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. A demonstration of an acceptable ICV is required.

16.6.4 Continuing Calibration Verification (CCV)

Acceptance Criteria

- The percent difference (%D) must be ≤30% (single analyte or hydrocarbon range). If more than one compound fails to meet this criteria, or any one analyte or range is >50% then the CCV is considered unacceptable.
- The relative retention time (RRT) and RRT window for each target APH analyte, internal standard, and hydrocarbon range "marker" compound must be verified with each CCV analyzed.

Corrective Action – If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it still fails prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects may be reported with the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative. Otherwise the sample affected by the unacceptable CCV shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

16.6.5 Method Blank

Acceptance Criteria – The method blank result for any target analyte must not be greater than the reporting limit and should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.

Corrective Action – If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results

are acceptable.

If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). Regardless, appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 18.3.

16.6.6 Laboratory (Sample) Duplicate

Acceptance Criteria – The relative percent difference (RPD) must be <30% when the results are >5x the MRL.

- If the RPD exceeds 30 and both results are >5x the MRL, the sample analysis must be repeated.
- If an analyte is detected in one analysis at >5x the MRL but not detected in the duplicate analysis, the analysis must be repeated.
- If an analyte is detected in one analysis at ≤5x the MRL but not detected in the duplicate analysis, the RPD is not calculable and the analysis does not have to be repeated.

Corrective Action – If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.6.7 Internal Standards

Acceptance Criteria – Internal standards must be adequately resolved from individual compounds in the APH calibration standard. A minimum separation requirement of 50% (maximum peak height to valley height) must be met, particularly for n-hexane and bromochloromethane (IS1). The internal standard area counts of each sample, blank, and Laboratory Control Sample must be evaluated against the corresponding continuing calibration standard or the midlevel initial calibration standard (if analyzed in the same sequence). The internal standard area counts must be within 50-200% of the continuing calibration standard area counts. If the internal standards fall outside this range, the sample, blank, or Laboratory Control Spike must be reanalyzed.

Corrective Action – If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If the problem is corrected with the lower volume analysis, report those results.

16.6.8 Sample Analysis

Acceptance Criteria – Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.

- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria.

- All target analyte peaks must be within the initial calibration range or reported with the appropriate data qualifier.
- The internal standard with each sample must comply with the requirements listed in Section 16.6.7.
- Each analyte, in order to be reported, must meet the qualitative identification requirements listed in Section 12.9.1.

Corrective Action – When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.7 Sample's Holding Time Expired The customer is to be notified that the sample's holding time was missed and the customer is to decide if the sample analysis is to continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual.

Note: No analyte results may be reported with an unacceptable initial calibration or initial calibration verification standard. However, any analyte not meeting such requirements (and the initial calibration is to be used) must be eliminated from the reporting list and any action taken fully documented.

18.2 Continuing Calibration Verification

- When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
- If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects may be reported with the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative. If this is the case then a full explanation must be noted in the case narrative of the final report. Refer to Section 15.5 for additional reporting requirements.

18.3 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

18.4 Laboratory Control Sample All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

18.5 Laboratory Duplicate All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

18.6 Internal Standard All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

18.7 Estimated Sample Results

18.7.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this standard operating procedure.

18.7.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

18.7.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported has having less certainty by reporting with the appropriate data qualifier.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use.

19.2 Method Detection Limit (MDL) This method does not require that MDL studies be performed. However, the APH target compounds are a subset of the EPA TO-15 analysis for which the laboratory performs MDL studies. The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 of the *SOP for Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS)* and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

19.3 Accuracy and Precision Refer to Section 16.5.1 above for information on replicate precision and accuracy criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets

the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the TO-15 Method Manual.

19.4 Selectivity Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 98 or newer version
- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

19.5 Demonstration of Capability

See Sections 16.5 and 16.6 for initial and ongoing method QC requirements.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
09.0	03/21/15	C. Humphrey	Section 4 – Revised section to include Hazard Assessment table
			Section 12.9 – Added Note 2
			Attachment 3 – Replaced Data Review Checklist with combined TO-15/MAPH Daily QC and Sample Review Checklists

21) References and Related Documents

21.1 *Method for the Determination of Air-Phase Petroleum Hydrocarbons (APH)*, Final Revision 1, Massachusetts Department of Environmental Protection, December 2009.

21.2 *SOP for Batches and Sequences*, SOP ID ADM-BATCH_SEQ

21.3 *SOP for Making Entries onto Analytical Records*, SOP ID CE-QA007

21.4 *SOP for the Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*, SOP ID CE-QA011

21.5 *SOP for Manual Integration Policy*, SOP ID CE-QA002

21.6 *SOP for Nonconformance and Corrective Action*, SOP ID CE-QA008

21.7 *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters,* SOP ID SMO-CanCert

21.8 *SOP for Flow Controllers and Critical Orifices,* SOP ID SMO-Flow_Cntrl.

21.9 *SOP for Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS),* SOP ID VOA-TO15

21.10 2009 TNI Standards

22) Appendix

22.1 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Daily QC and Sample Review Checklists

Attachment 1

Training Plan

Training Plan for Analysis of Air-Phase Petroleum Hydrocarbons (APH) by GC/MS

SOP Title: _____ Revision:_____ Date: _____

Trainee: _____ Trainer: _____ Instrument: _____

1. Read SOP — Trainer ____ Trainee ____ Date _____

2. Read Method — Trainer ____ Trainee ____ Date _____

3. Demonstrated understanding of the scientific basis of the analysis — Trainer ____ Trainee ____ Date _____
   Training Duration _____
   - Whole air sample preconcentration techniques
   - Gas chromatography
   - Mass spectrometry

4. Demonstrated familiarity with related SOPs — Trainer ____ Trainee ____ Date _____
   Training Duration _____
   - SOP for Batches and Sequences
   - SOP for Making Entries onto Analytical Records
   - SOP for Manual Integration Policy
   - SOP for Significant Figures
   - SOP for Nonconformance and Corrective Action
   - SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation
   - SOP for Cleaning and Certification of Summa Canisters

5. Observe performance of SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   - ___sample preparation/dilution and sample loading and analysis
   - ___analytical sequence setup
   - ___standard preparation
   - ___BFB tuning evaluation/initial calibration/initial calibration verification
   - ___continuing calibration verification
   - ___EnviroQuant introduction
   - ___data reduction and reporting
   - ___canister handling

6. Perform SOP with supervision *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   - ___sample preparation/dilution and sample loading
   - ___analytical sequence setup
   - ___standard preparation
   - ___BFB tuning evaluation/initial calibration/initial calibration verification
   - ___continuing calibration verification
   - ___sample analysis
   - ___EnviroQuant use
   - ___data reduction and reporting
   - ___canister handling

7. Independent performance of the SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   - ___sample loading and sample dilutions
   - ___analytical sequence setup
   - ___standard preparation
   - ___BFB tuning evaluation/initial calibration/initial calibration verification
   - ___continuing calibration verification
   - ___sample analysis
   - ___EnviroQuant proficiency
   - ___data reduction and reporting
   - ___canister handling
   - ___initial demonstration of competency
     - -4 Laboratory Control Samples

8. Instrument operation and maintenance *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   - ___autosample
   - ___GC and capillary column installation
   - ___mass spectrometer
   - ___data system

Attachment 2

Initial Calibration Checklist

Method: **MAPH**
ICAL Date: _____
Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS____

Air-Phase Petroleum Hydrocarbons
Initial Calibration Review Checklist

Analyst **Reviewer**

☐ 1. Is the required documentation in the ICAL file? ☐
- ☐ BFB Tune analysis Report ☐
- ☐ Calibration Status Report (aka Calibration History) ☐
- ☐ Response Factor Report/Percent RSD (target analytes) ☐
- ☐ Percent RSD Report (hydrocarbon ranges) ☐
- ☐ Quantitation Report for each calibration standard (including manual integration documentation) ☐
- ☐ ICV Quantitation Report ☐

☐ 2. Was the ICAL performed continuously (i.e., not interrupted for maintenance or for sample analysis)? ☐

☐ 3. Have all the calibration standards been analyzed within 24 hours of each other? ☐

☐ 4. Does the BFB tune check standard analysis at the start meet the tune criteria? ☐

☐ 5. Are all the analytes in the blank analysis <MRL? ☐

☐ 6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels? ☐

☐ 7. Were the standards analyzed from low concentration to high concentration? ☐

☐ 8. For each analyte or range, are there no levels skipped? ☐

☐ 9. For each analyte or range, is there only one value used for each calibration level? ☐

☐ 10. For each analyte range, is the lowest standard's concentration at or below the analyte's MRL? ☐

☐ 11. If a calibration level is dropped, are all the responses for each target analyte and range dropped and is the information noted in the ICAL explaining the reason? ☐

☐ 12. Is the average RSD ≤30% for all analytes and ranges, except *naphthalene* can be ≤40%? ☐

☐ 13. For the ICV analysis, are all the analytes within 70%-130% recovery? ☐

☐ 14. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages. ☐

COMMENTS:

Analyst: _____ Date: _____

Secondary Reviewer: _____ Date: _____

Attachment 3

Daily QC and Sample Review Checklists

Daily QC Review Checklist

(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

EPA Compendium Method TO-15

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		**Reviewer**
☐	1. Is the required documentation present? ☐ CORRECT BFB Tune analysis Report ☐ CCV analysis Quantitation Report & %D Report ☐ LCS analysis Quantitation Report ☐ MB analysis Quantitation Report	☐
☐	2. BFB **tune** check standard analysis meet the tune criteria for the method indicated above?	☐
☐	3. Analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**?	☐
☐	4. Does the **CCV** have a difference ≤30% for all analytes? [Note all outliers biased high and/or low]	☐
☐	5. All **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)?	☐
☐	6. All **IS** responses within ±40% of CCV or the midpoint in the ICAL?	☐
☐	7. All **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%)	☐
☐	8. All analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)?	☐
☐	9. **LCS** %R within the lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)?	☐
☐	10. All analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits?	☐

COMMENTS:

Air-Phase Petroleum Hydrocarbons

☐	1. Does the **CCV** meet the following criteria? • Percent difference ≤30%. • One compound or range can be >30%, but less than 50%. • No single analyte or range may be >50%. [Note outliers biased high and/or low]	☐
☐	2. Does lab **duplicate** meet an RPD of ≤30% for results >5x MRL? Repeat analysis if:	☐
☐	3. Are the analytes in the **LCS** within 70%-130% recovery?	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐ LIMS Run Approval ☐ LIMS Supervisor Approval

Analyst: _____ Secondary Reviewer: _____

Date: _____ Date: _____

Sample Review Checklist
(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

EPA Compendium Method TO-15

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____ **Project #:** _____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. All analyte hits in the samples within the **calibration range** and/or noted?	☐
☐	2. All **peak integrations** acceptable?	☐
☐	3. All **manual integrations** flagged and documented?	☐
☐	4. Have **Q values** been verified for each peak?	☐
☐	6. All **calculations** correct?	☐
☐	7. Has the analyst initialed and dated each **quantitation report**?	☐
☐	8. For **TICs** are the relative intensity and other requirements met?	☐
☐	9. **Auto report** correct?	☐
☐	10. **MRL** = _____ ☐ ng ☐ pg (ethanol, acetone, vinyl acetate = 5.0ng)	☐
☐	11. Pressurized with **Helium**? Is the worksheet completed for all samples?	☐
☐	12. Report to **MDL**? ☐ Yes ☐ No	☐
☐	13. **Global Minimum Detection Limit** = _____ ☐ ng ☐ pg	☐
☐	14. **DOD:** Are **manual integrations** notated in the **case narrative?**	☐

COMMENTS:

Air-Phase Petroleum Hydrocarbon

☐	1. Are all manual **integrations** flagged and documented (except for HC ranges)?	☐
☐	2. Are all peak **integrations** acceptable?	☐
☐	3. Has the analyst initialed and dated each **quantitation report**?	☐
☐	4. Are the associated ICAL responses correct?	☐
☐	5. Are the sample responses entered into the template correctly?	☐
☐	6. Are the TO-15 target compounds entered into the template correctly?	☐
☐	7. Does the lab **duplicate** meet a RPD of ≤30% for results >5x the MRL? Otherwise, repeat analyses if:	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐ LIMS Run Approval

Analyst: _____

Date: _____

☐ LIMS Supervisor Approval

Secondary Reviewer: _____

Date: _____

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCED METHOD:	EPA TO-15
SOP ID:	VOA-TO15
REV. NUMBER:	22.0
EFFECTIVE DATE:	03/21/2015

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STANDARD OPERATING PROCEDURE

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

EPA TO-15

SOP ID:	VOA-TO15	Rev. Number:	22.0	Effective Date:	03/21/2015

Approved By: Wida Ang — Date: 3/13/15
Team Leader (VOA GC/MS) - Wida Ang

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QA Manager - Chaney Humphrey

Approved By: Kelly Horiuchi — Date: 3/13/15
Laboratory Director - Kelly Horiuchi

Archival Date: _____ Doc Control ID#: Non-Controlled Editor: _____

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DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1) Scope and Applicability

1.1 This procedure is based on and incorporates the requirements detailed in EPA Compendium Methods TO-15 and TO-14A and is used to quantify a wide range of volatile organic compounds (VOCs) in gaseous matrices collected in gas collection bags (method modification) and specially prepared stainless steel canisters or glass bottles. This method typically applies to ambient concentrations of VOCs 0.50ug/m3 (down to 0.10ug/m3 for low level ambient analyses) and above for the SCAN mode and 0.010ug/m3 and above for the SIM mode; however, refer to Tables 3 and 3A for the specific laboratory initial calibration ranges for each target compound. The method requires VOC enrichment by concentrating up to one liter of a sample volume, with a virtually unlimited upper concentration range using dilutions from source level samples.

In this document, Tables 2 and 2A (see Note 1 below) list compounds that can be determined by this procedure along with their corresponding laboratory method reporting limits (MRLs) and method detection limits (MDLs). The reported MRL may be adjusted higher; however, the capability of achieving lower MRLs for specific project requirements must be thoroughly demonstrated (by an acceptable initial calibration and method reporting limit check standard) and documented as long as the MRL is higher than the current method detection limit for each compound. Additional compounds may be analyzed according to this procedure as described in the referenced methods as long as the requirements of this document are adhered to; however, if a compound is not listed in the TO-15 method, refer to Note 1 below. The number of samples that may be analyzed in a 24-hour period is about twenty. The number of sample results that may be reduced in an eight-hour day is approximately twenty.

2) Summary of Procedure

2.1 The analytical method involves using a high-resolution gas chromatograph (GC) coupled to a mass spectrometer (MS). The GC/MS utilizes a linear quadrupole system, which allows for it to be operated by either continuously scanning a wide range of mass to charge ratios (SCAN mode) or by Select Ion Monitoring mode (SIM), which consists of monitoring a small number of ions from a specified compound list.

An aliquot of an air sample is concentrated on a solid adsorbent trap (either cryogenically or fan cooled glass beads or stronger adsorbents at higher temperatures) to collect the analytes of interest. To remove co-collected water vapor, the concentrated sample then goes through a water removal (dry purge) step. After the sample is pre-concentrated on a trap, the trap is heated and the VOCs are thermally desorbed onto a refocusing cold trap. The VOCs are then thermally desorbed onto the head of a capillary column once the cold trap is heated. The oven temperature (programmed) increases and the VOCs elute and are detected by the mass spectrometer.

Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the

system response to the primary fragment for known amounts of the compound. This method utilizes the internal standard calibration technique; refer to Section 3.16 for a complete definition.

3) Definitions

3.1 Cryogen A refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Liquid nitrogen (cryogen) is used for this purpose and it has a boiling point of -195.8°C.

3.2 Gauge Pressure Pressure measure with reference to the surrounding atmospheric (barometric) pressure, usually expressed in units of psig. Zero gauge pressure is equal to atmospheric pressure.

3.3 MS-SCAN Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

3.4 MS-SIM Mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].

3.5 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.6 Neat Stock Standard A purchased, single component assayed reference material having a stated purity used to prepare working calibration standards.

3.7 Stock Standards Solution A concentrated solution of one or more target analytes at a known concentration purchased from a reputable commercial vendor. Stock standard solutions are used to prepare working calibration standards.

3.8 Intermediate Calibration Standard A solution of one or more target analytes at a known concentration prepared either from one or more neat stock standards or from one or more stock standards solutions.

3.9 Working Calibration Standard A solution of all the target analytes at a known concentration prepared either from one or more intermediate calibration standards and/or from one or more stock standard solutions.

3.10 Calibration or Standard Curve A calibration or standard curve is a graph which plots the concentration of a compound (or an analyte) versus the instrument response to the compound.

3.11 Initial Calibration Verification (ICV) Standard A solution prepared in the laboratory containing known concentration(s) of analytes of interest. The solution is prepared from neat stock standards and/or stock standards solutions which are from a different source than the standards used to prepare the working calibration standards.

3.12 Continuing Calibration Verification (CCV) Standard A working calibration standard which is analyzed at specific intervals in order to verify that the instrument continues to meet the calibration criteria.

3.13 Field Sample A sample collected and delivered to the laboratory for analysis.

3.14 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop "ticks" have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.15 Batch Quality Control (QC) Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD).

3.16 Internal Standard Calibration Compares the instrument responses from the target compound in the sample to the responses of specific standards (called internal standards), which are added to the sample or sample preparation prior to analysis. The ratio of the peak area (or height) of the target compound in the sample or sample preparation is compared to a similar ratio derived for each calibration standard.

3.17 May This action, activity, or procedural step is neither required nor prohibited.

3.18 Must This action, activity, or procedural step is required.

3.19 Shall This action, activity, or procedural step is required.

3.20 Should This action, activity, or procedural step is suggested, but not required.

3.21 SOP Standard Operating Procedure

3.22 Service Request A form generated, at the time of sample receipt, which details pertinent information such as client name, address, contact, client and laboratory sample identifications, sampling and receipt dates and times, requested analyses, sample type, canister pressures (initial and final), and the service request number (unique number for each submitted job) and serves as an inter-laboratory "custody" form which accompanies all samples throughout the laboratory.

3.23 Selectivity Selectivity of a method refers to the extent to which it can determine particular analyte(s) in a complex mixture without interference from other components in a mixture. Another definition is the extent to which a particular method can be used to determine analytes under given conditions in the presence of other components of similar behavior.

3.24 Limit of Detection (LOD) The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DoD Clarification). For consistency purposes, the LOD may be referred to as the MDL once it is reported; however, full verification will be on file in the laboratory per the procedures detailed in this document.

3.25 Limit of Quantitation (LOQ) The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. (DoD Clarification). For consistency purposes and since the LOQ and MRL are equivalent with regards to laboratory procedure, the LOQ will be referred to as the MRL in this document and once it is reported. Full verification will be on file in the laboratory per the procedures detailed in the document.

3.26 Detection Limit (DL) / Method Detection Limit (MDL) The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type 1 error) is 1%. (DoD Clarification). For consistency purposes, the DL may be referred to as MDL. Also, as far as reporting is concerned the MDL will be raised up (where necessary) to the verified LOD per the procedures defined in this document and reported accordingly.

4) Health and Safety Warnings

4.1 Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, Safety Data Sheet (SDS) location, and the laboratory waste management plan for the safe disposal of chemicals and samples.

4.2 Pollution Prevention and Waste Management

All waste disposals shall be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal.* In addition, canisters must be cleaned in accordance with the requirements detailed in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters.*

4.3 This procedure may include CHEMICAL, OPERATIONAL and/or EQUIPMENT hazards. Employees must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1: Standard and Sample Preparation	**Hazards**	**Preventative Measures**
Compounds, mixtures of compounds, standards, surrogates, and samples.	Exposure to potential health hazards through absorption through skin. Inhalation hazards.	Reduce exposure through the use of gloves and fume hoods. Safety glasses must be worn when working in the prep lab. Care should be taken when handling standard material in a neat or highly concentrated form. Personal protective clothing (safety glasses, gloves, and lab coat) are required when handling standard material in neat form. Consult Safety Data Sheets (SDS) for compounds being handled in this procedure, and be familiar with proper safety precautions.
Job Task #2: Working with Liquid Nitrogen	**Hazards**	**Preventative Measures**
Turning valves and handling tubing and fittings that have been in contact with the cryogen.	Can cause serious tissue damage (frostbite) with only a few seconds of contact.	Wear neoprene or leather gloves. Valves on cryogen dewars should be opened slowly so leaky fitting can be identified.
Job Task #3: Working with Pressurized Gases	**Hazards**	**Preventative Measures**
Using and moving compressed gas cylinders.	Gas leak, fire, and explosion. Personal injury due to falling during transport.	All cylinders must be secured in an upright position to a wall or immovable counter with a chain or a cylinder clamp when not in use. Keep safety caps on when cylinders are not in use. A handcart must be used when transporting cylinders. The cylinder must be secured to the handcart with a chain or belt. The regulator should never remain on small "D" size cylinders following use. Full cylinders must be kept separate from empty cylinders. Flammable gases (i.e. pressurized hydrogen) must be clearly labeled. Flammables and oxidizers must be separated by a ½-hour fire wall or by at least twenty feet.
Job Task #4: Glass Syringes	**Hazards**	**Preventative Measures**
Glass syringe use	Skin lacerations and punctures.	The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

5) Cautions

5.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

The instrument maintenance log must be kept current. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The entry in the log must include.

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful tune and continuing calibration or initial calibration and the file number that accompanies the data will serve as proof that the maintenance is complete and the instrument is in working order.

The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, changing the pump oil, cleaning the source, ordering a part. In addition, a notation should be made in the logbook stating that no samples were analyzed during the days that the instrument was down and no active maintenance was being conducted (i.e., where no other notation was made in the logbook for those days).

5.2 Concentrating Trap

Routine maintenance includes periodic solvent cleaning of the Silco steel lines in the valve oven if contamination is suspected. Also, periodic replacement of the multi-sorbent or partial replacement of the trap if analyte specific deterioration is detected is required. For specific trap information refer to the instrument maintenance logbook and electronic method manual.

After repacking, the trap should be baked at 265°C for a minimum of two hours (or until a clean blank is generated) and a partial repacking requires baking (at 265°C) the trap for a minimum of 20 minutes (or until a clean blank is generated).

5.3 GC System

Column performance is monitored by observing both peak shapes and column bleed. Over time, the column will exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced (see Section 9.5). Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Declining performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.4 Mass Spectrometer

The Mass Selective Detector (MSD) ion source requires periodic cleaning to maintain proper performance. Symptoms of a dirty ion source include difficulty keeping the MSD in tune and fluctuating internal standard areas. The vacuum system should be serviced every six months, including changing the pump oil and checking the molecular sieve in the back-streaming trap.

5.5 Instrument Tuning

The instrument is tuned with guidance from the procedure described in the HP Operations Manual, when necessary.

5.6 Computer Troubleshooting

Computer care and troubleshooting is conducted by the IT department. Refer to Section 9.6 for the computer hardware and software requirements.

Computers are selected to meet or exceed operating system and or acquisition software requirements. Periodic upgrades of memory are performed to maintain or improve system performance and reliability. Upgrades may be performed on systems until instrument hardware configurations become the limiting factor.

Basic Troubleshooting Outline:

1) Document occurrence and severity in IT Log
2) Interview user(s)
3) Investigate any available logs (Event Logs, Acquisition Logs, etc.)
4) Determine if problem is isolated (single user or acquisition) or widespread (multi user or network).
5) If multiple possibilities exist for cause, then eliminate in systematic manner.
6) Hardware issues are addressed with component replacement (beginning with most suspect portion).
7) Software issues are addressed first with internet investigation (user blogs, software source updates/findings).
8) Network issues are investigated from the Server, to Switch, to Network Card; utilizing all available managed devices to help discover possible failure points.
9) In some cases, system corruption may require reload or complete system replacement.
10) Finalize documentation in IT Log with actions taken
11) Perform periodic follow-up with User and review any log found to have suspect events that suggested source of issue.

6) Interferences

6.1 Summa Canisters

Canisters shall be stored in a contaminant free location and shall be capped tightly during shipment to prevent leakage and minimize any compromise of the sample. The pressure/vacuum is checked prior to shipment and upon receipt from the field. Any problems with the sample from the field are noted and the Project Manager contacted.

Also, canisters must be cleaned and certified to be free from target analytes before being shipped to the field for sample collection. The procedure is described in detail in the *SOP for Cleaning and Certification of Summa Canister and Other Specially*

Prepared Canisters (refer to this procedure as well as Section 16.7 for the acceptance criteria).

Current laboratory practice entails the segregation of 6L canisters into ambient (low) level and source levels. All the ambient canisters are used for low level (indoor air, ambient air) projects and not intentionally for soil gas, SVE monitoring, or other higher level applications. It may be necessary to "retire" an ambient canister and re-assign for source level use if high concentrations are encountered. This decision will be made by management based on analytical concentrations and what compounds were encountered at these levels. If the level of any analyte is detected above 5,000ug/m*3* in the ambient can, then the supervisor/team leader must be contacted to determine if the canister(s) is to be retired. If retirement is decided upon, make a notation on the sample tag (or other color coded tag) of each canister in question. The notation must contain the analyte, threshold levels and retirement from ambient use (initial and date notation) so that the canister conditioning/management department may properly execute the retirement.

6.2 Analytical System

The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with buna-N rubber components must be avoided.

6.3 Carbon Dioxide

Excessive levels of carbon dioxide present in a sample may interfere with analysis by freezing up the cryogenic trap. A smaller aliquot must be analyzed to eliminate this problem, or the sample should be analyzed using the higher temperature multi-adsorbent trapping technique which allows carbon dioxide to pass.

6.4 Gas Collection Bags

This procedure covers the use of gas collection vessels such as Tedlar® or Mylar® bags. However, due to the nature of these types of bags it is not recommended that clients use this option for ambient air samples. Sample collection bags made out of ®Tedlar have contaminants that are inherent to the manufacturing process. The two main contaminants are phenol and N,N-Dimethylacetamide. However, this only becomes a problem when the concentration levels in the sample are low ppbv such as ambient air monitoring samples where more of the sample usually has to be concentrated and analyzed. To minimize the loss of sample integrity, a 72-hour hold time has been incorporated into the procedure.

6.5 Glassware

Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware results in discrete artifacts and/or elevated baselines in the detector profiles should be minimized. All glassware associated with this method must be scrupulously cleaned to avoid possible contamination. The cleaning shall be performed in accordance with the procedure outlined in the *SOP for Glassware Cleaning*. The use of high purity water, reagents, and solvents helps to minimize these problems.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP

may perform analysis, interpretation and peer review of the results. Data reduction and/or peer review may be performed by another qualified employee. This employee must be familiar with the analytical technique and have completed a data review training plan to ensure familiarity with specific analysis and requirements.

7.2 The supervisor/manager must ensure that method proficiency is documented initially and whenever significant changes in the instrument type, personnel, and matrix or test method are made.

7.3 The department supervisor/manager or designee shall perform final review and sign-off of the data.

7.4 Demonstration of Capability

All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy*. Demonstrations shall also be performed in accordance with the 2009 TNI Standards (Volume 1 Module 4 Section 1.6) and DoD Quality Systems Manual 5.0. Attachment 1 shall be used to document the training plan for new analysts' initial demonstration. Additionally, these demonstrations are performed anytime there is a change in instrument type, personnel or method.

Once performance is found to be acceptable, a required certification statement must be completed by the QA Manager and either the immediate supervisor or Laboratory Manager and retained on file as a demonstration of compliance.

7.4.1 Quarterly Demonstration A demonstration of method sensitivity must be performed *quarterly on each instrument* performing this method.

1) A spike at the current LOD must be analyzed.

2) Verification of precision and bias at the LOQ must be performed.

Refer to Section 11.1.4.2 (LOQ) and 12.14.1 (LOD) for additional information on how these demonstrations are to be performed as well as the acceptance criteria.

7.4.2 Annual Demonstration Each analyst must perform this demonstration both initially and annually. Analyze four LCS standards at 1-4x the MRL (LOQ) either concurrently or over a period of days as a verification of precision and bias of the quantitation range. The standard deviation (n-1) and average percent recovery of the four replicates are compared against the method requirement for precision (±25%) and current laboratory control limits for bias/LCS.

7.4.3 Change in Personnel, Instruments, Method and/or Matrix The requirements in Sections 7.4.1 and 7.4.2 must be performed per the schedule noted and when there is a change in personnel, instruments, method or matrix. "Change" refers to any change in personnel, instrument, test method, or sample matrix that potentially affects the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, or other components of the sample analytical system, or a method revision).

All completed attempts at this demonstration must be completed and turned into the QA department for retention.

8) Sample Collection, Handling, and Preservation

8.1 Air samples are collected in the field and delivered to the laboratory and shall be collected in either a specially prepared, leak-free, stainless steel pressure vessel (with valve) of desired volume (e.g., 6L), a glass sampling bottle (Bottle Vac, Entech

Inntruments) or a sample collection bag (Tedlar). Canister samples may either be grab or time integrated (using a variable flow controller, refer to the *SOP for Flow Controllers and Critical Orifices*) utilizing the canister vacuum to draw the sample. Bags require the use of an upstream pump or a "lung machine."

8.2 There are no special preservation requirements for either canisters, Bottle Vacs or bags. However, bags should be stored in an environment free from puncture or deterioration sources (by hanging them from clips), labeled with the specific service request number, in accordance with the *SOP for Laboratory Storage, Analysis and Tracking*. Canisters and bottles should be stored on the appropriate shelves until they are to be analyzed.

8.3 Sample collection bags must be analyzed within 72 hours from the confirmed time of sampling. Samples received by the laboratory shall be analyzed within 30 days of sampling or sooner if project specific requirements dictate. Programs, which have shorter recommended or required hold times, include the Department of Toxic Substances Control (DTSC), which advises a 72 hour hold time. The Minnesota Pollutions Control Agency (MPCA) and EPA Region 9 both require a 14 days hold time. Additionally, the MPCA does not allow the use of Tedlar bags for sampling or sample dilution. The DTSC requirement is an advisory notice, but the laboratory shall make every effort to comply. However, the following statement shall be added to each report where sample analyses do not meet the 72 hour hold time and the client project is intended to comply with DTSC requirements. "The recommended 72-hour hold time for the analysis of TO-15 was exceeded per the DTSC and LARWQCB Advisory – Active Soil Gas Investigations document dated January 28, 2003; however, this specific hold time statement is advisory and not considered as regulation. In addition, the samples were analyzed within the EPA Method TO-15 stated requirement of 30 days."

9) Equipment and Supplies

9.1 Additional instruments and/or differing models may be utilized as long as they are equivalent and meet the minimum requirements of this document.

9.2 Gas Chromatograph (GC)

An instrument capable of temperature programming, with a column oven that may be cooled to sub-ambient temperature at the start of the gas chromatographic run to result in the resolution of the VOCs.

Hewlett Packard 5890 Series II Plus
Hewlett Packard 6890 Series
Hewlett Packard 6890A Series
Agilent 6890N Series
Agilent 7890A Series

9.3 Autosampler

Tekmar-Dohrmann AUTOCan Autosampler:	14-ACAN-074
Concentrating Trap (cryogenic trap, built-in):	14-6938-020
Cryofocusing Module w/split valve:	14-6520-A00
GAST Vacuum Pump:	DOA-P104-AA or equivalent

9.4 Mass Spectrometer (MS)

A MS capable of scanning from 34 to 350 amu every second or less, using 70 volts

(nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

Hewlett Packard 5972 Series
Hewlett Packard 5973 Series
Agilent 5973N
Agilent 5973 *inert*
Agilent 5975B *inert*
Agilent 5975C *inert*

9.4.1 Ionization Gauge Controller

- Agilent: 59864B
- Granville-Phillips 330 Ionization Gauge Controller: 330001/2/3
- Hewlett Packard Ionization Gauge Controller: 59864B

9.5 Analytical Column

Any analytical column capable of separating the compounds of interest may be used. The capillary column should be directly coupled to the source of the mass spectrometer. The following are suggested columns; an alternative column may be used as long as sufficient peak resolution and separation is achieved.

- Restek Rxi-1ms Fused Silica Capillary Column; 30m x 0.25mm ID 1.0µm film thickness

  OR

- Restek Rxi-1ms Fused Silica Capillary Column; 60m x 0.25mm ID 1.0µm film thickness

9.6 Data Systems

IBM-compatible PC with Windows 95/98/NT/XP/7 (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library (2002 version or newer) or equivalent.

9.7 Canister Pressurization Station

Vacuum/Pressure Gauge [0 to –30 inHg; 0-90 or 100 psig]

9.8 Canister Sampling Devices

Refer to the *SOP for Flow Controllers and Critical Orifices* for specific calibration and other pertinent information.

- VICI Condyne Model 300 Flow Controller
- Critical Orifices (Laboratory manufactured)

9.9 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent
- Lab Commerce, Stabilizer Model 22.4L, 2.4L Canisters or equivalent
- Restek Corporation, #24203, 3.0L Silco Canisters or equivalent

- Tedlar bags – 0.5L, 1L, 3L, 5L, 10L, 25L, and 40L (other sizes are available; however, the volumes that are listed encompass the majority of the bags supplied and the samples submitted to the laboratory).

9.10 Dynamic Dilution System

- Entech Dynamic Diluter Model 4620A
- Toshiba laptop computer Model 2210CDT/6.0 and Software NT460

10) Standards and Reagents

10.1 Reagents and Equipment

10.1.1 UHP Grade Helium (99.999%) (GC carrier gas, preconcentrator purge/sweep gas, pressurization gas)

10.1.2 Cryogen - Liquid nitrogen from bulk tank or 50 psig dewars (used to cool preconcentrator traps)

10.1.3 UHP/Zero Grade Air (canister pressurization)

10.1.4 ASTM Type II Water, DI water or equivalent

10.1.5 UHP Grade Nitrogen (99.999%) (additional pressurization gas, based on other methods requested – modification to method)

10.2 Standards

Standards are prepared for both SCAN and Selective Ion Monitoring (SIM) modes according to the procedures detailed in this section. The preparation of standards for the analysis of air samples is carried out by following the procedure, "Preparation of Gas Phase Standards for Ambient Air Analysis", Application Note, Spring 96, Vol. 6.5, *Tekmar*-DOHRMANN AutoCan User's Manual. Neat standards that are used for making trace gas standards must be of high purity; generally a purity of 98 percent or better is commercially available.

10.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Mixture Prepare a standard solution of p-Bromofluorobenzene (BFB-used as both a tune check and surrogate compound), bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene, 1,2-dichloroethane-d4(surrogate), and toluene-d8(surrogate) at 500µg/m³ each in humidified zero air (Section 9.2.1.2). Prepare this standard according to the procedure outlined in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note. This standard may also be prepared from a neat cocktail as in Section 10.2.2.2.1 or as stated in Section 10.2.1.3.

10.2.1.1 An intermediate standard is prepared from neat compounds in a glass static dilution bottle (SDB). After the volume of the SDB is determined, calculate the mass of each compound to be spiked to achieve a final concentration of 5.0µg/ml. Then use the density of each neat compound to calculate the microliter amount to be spiked into the SDB. The SDB is then heated for a minimum of one hour at ~60°C to completely volatilize all components.

Concentration of the intermediate standard prepared in a SDB is 5.0µg/mL. The amount required to achieve this concentration is determined through the use of the following equation.

$$A = \frac{(C)(V)}{D} \qquad \text{(Equation 1)}$$

Where:

A Amount of each compound required to achieve the desired concentration of the standard in the SDB (µL)
C Desired concentration of SDB (µg/mL)
V Actual volume of the SDB (mL)
D Density of the compound in question (µg/µL)

Example:

Calculate the amount of neat bromochloromethane needed to achieve the final concentration of 5.0µg/mL of that compound in the SDB.

V = 2010mL
D = 1934.4µg/µL
C = 5.0µg/mL

$$A = \frac{\left(5.0\frac{\mu g}{mL}\right)2010mL}{1934.4\frac{\mu g}{\mu L}} = 5.2\mu L$$

Density (µg/µL)	Compound
1934.4	Bromochloromethane
1170.1	1,4-Difluorobenzene
1157	Chlorobenzene-d5
1307	1,2-Dichloroethane-d4
943	Toluene-d8
1593	BFB

10.2.1.2 The Working standard is prepared in a Summa canister by spiking an aliquot of the stock SDB standard (Section 10.2.1.1) using a heated gastight syringe. Connect a cleaned, evacuated Summa canister to a source of pure diluent gas (humidified zero air) using a Teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum. Spike the SDB stock and following removal of syringe a small flow of diluent gas to flush the spike into the can. Pressurize the can to positive 83.3 psig with humid zero air, and allow the contents to equilibrate for approximately 24 hours before using.

Concentration of the working standard prepared in a Summa canister is 500ng/L. The final pressure of the canister is 83.3psig; therefore, the pressurized volume is 40L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 2)

Where:

PV Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PF = $\frac{P_{atm}+P_f}{P_{atm}+P_i}$

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister at 1atm

P_{atm} Atmospheric Pressure = 14.7psig

Example:

$\frac{14.7+83.3}{14.7+0}(6L)$ = 40L

In order to prepare the canister with a concentration of 500ng/L, it must be determined how much of the intermediate standard is required. This is achieved through the use of the following equation.

$$A = \frac{(F)(V)}{(C)\left(1000\frac{ng}{\mu g}\right)} \qquad \text{(Equation 3)}$$

Where:

F Desired concentration of working standard (ng/L)

V Pressurized Volume of Canister (L)

C Concentration of prepared SDB (µg/mL)

A Amount of standard (mL) of the SDB required to obtain the desired working standard concentration

Example:

$$A = \frac{500\frac{ng}{L}(40L)}{\left(5.0\frac{\mu g}{mL}\right)\left(1000\frac{ng}{\mu g}\right)} = 4mL$$

10.2.1.3 Currently the working standard is purchased in a cylinder at a certified concentration of 500ng/L (prepared by Linde SPECTRA Environmental Gases, Alpha, NJ).

10.2.1.3.1 For SCAN analyses, the working standard is filled directly into a summa canister to a pressure of 70 to 80 psig.

10.2.1.3.2 For SIM analyses, the working standard is diluted and pressurized with humid zero air to the desired concentration

using Equation 2 in Section 10.2.1.2. Typical concentrations will be 20ng/L, 40ng/L or 50ng/L.

10.2.2 Initial Calibration (ICAL) Standard Prepare the primary source calibration standards in Summa canisters with nominal concentrations of 1ng/L (optional), 20ng/L and 200ng/L for analyses in SCAN mode and 0.1ng/L, 5.0ng/L, and 200ng/L for analyses in Selective Ion Monitoring (SIM) mode for each of the target analytes. Differing injection volumes will create the standard concentrations listed in Tables 3 (SCAN) and 3A (SIM) of this document. The full list of analytes which are analyzed according to this method can also be found in Tables 2 (SCAN) and 2A (SIM).

Standards are prepared by diluting the stock standard with humid zero air into a Summa canister. The stock standard is a certified custom-blended cylinder (prepared by Linde SPECTRA Environmental Gases, Alpha, NJ). Refer to Tables 3 and 3A for the list of analytes and certified concentrations in the purchased cylinder.

10.2.2.1 Working standards are prepared into Summa canisters using the Entech Dynamic Diluter. Turn on the power to the diluter one hour prior to using to allow for the components to come to thermal equilibrium. Connect the computer and start the software. Connect a Zero Air source to the humidification chamber (flow controller #1). Connect stock standard cylinder#1 to flow controller #2 inlet. Open the cylinder valves. Adjust the inlet pressures to 50 to 60psig.

Standard Concentration Selection: The concentration of the three working standards prepared in Summa canisters should be 200ng/L, 20ng/L and 1ng/L (depending on the dynamic range of the initial calibration include 1ng/L if a 0.08ng and 0.4ng on column standard is desired or this standard may be used for the 0.5ng/L concentration as well) for SCAN and 0.2ng/L, 4.0ng/L, and 200ng/L for SIM.

Position 1 – Total Air Flow (Zero Air)
Position 2 – Standard Flow (Purchased Standard One)
Position 3 – Standard Flow (Purchased Standard Two if Applicable)
Position 4 – Total Air Flow (Zero Air) (utilized if preparing a two dilution standard)
Position 5 – Diluted Standard Flow (utilized if preparing a two dilution standard)

Step1: Determine the required flow rate of the stock standards (positions #2 and #3). The range must be from 5 to 50sccm (standard cubic centimeters per minute, same as ml/min). The flows listed below are guidelines to be used for the default standard flow (based on the desired standard concentration) and were chosen based on the ultimate final dilution required and limitations of the Dynamic Diluter (flows must be from 150 to 2000ml/min.).

Desired Standard Conc.	Default Standard Flow
200ng/L	50ml/min
100ng/L	50ml/min
20ng/L	20ml/min
5.0ng/L	10ml/min
4.0ng/L	8ml/min

1ng/L	50ml/min; 20ml/min (See Note 1 below)
0.2ng/L	10ml/min; 20ml/min (See Note 1 below)

Note 1: For the 1ng/L and 0.2ng/L standards (or any standard requiring more than a 400X dilution of the stock), a slightly different procedure is performed. In order to prepare these standards, a double dilution must be performed which involves taking the primary dilution flow and making a secondary dilution of that using the diluent gas. Unscrew the cover of the dilutor and connect the first mass flow controller as well as the tubing to re-route the first dilution output from the final standard Summa canister to the 2nd dilution chamber. Refer to example 2 for the calculation guidelines to prepare a two dilution standard.

Example 1: Prepare a 200ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required dilution factor for each stock.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 200ng/L = 5

Step 3: Calculate Total Flow
Total Flow= (stock std. flow-see table above)*(Dilution Factor)
Total Flow=50ml/min*5 = 250ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=250ml/min-(50+50)ml/min = 150ml/min

Example 2: Prepare a 0.2ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required total dilution factor for the 0.2ng/L standard.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 0.2ng/L = 5,000

The two dilutions must be performed which total the dilution factor calculated above. Since the flow for the Diluter is restricted to a maximum of 2000ml/min, the total flow (as calculated in Step 3 below) cannot exceed 2000ml/min; therefore, the dilutions must be chosen accordingly.

Step 3: Calculate Total Flow
Total Flow = (stock std. flow-see table above)*(Dilution Factor)
Total Flow (Dilution 1) = 10ml/min*200 = 2000ml/min

For the 2nd dilution take the stock standard flow selected for dilution 1 for the two purchased cylinders (10ml/min each based on the desired final concentration) and add them together (10ml/min + 10ml/min for 20ml/min) to get the stock standard flow for the 2nd dilution.

2nd Dilution Factor Needed = Total Dilution/1st Dilution
2nd Dilution Factor = 10000/200(1st dilution) = 50

Total Flow (Dilution 2) = 20ml/min*50 = 1000ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=2000ml/min-(10+10)ml/min = 1980ml/min (Dilution 1)
Air Flow=1000ml/min-20ml/min = 980ml/min (Dilution 2)

Position 1 = 1980ml/min
Position 2 = 10ml/min
Position 3 = 10ml/min
Position 4 = 980ml/min
Position 5 = 20ml/min

Step 5: Enter flow rates in the appropriate fields in the Entech software. Start flows by clicking the "GO" button in the top right of the window. Allow flows to equilibrate for at least fifteen minutes, then attach an empty canister to the outlet port and open the valve. The outlet pressure will be displayed in the lower right of the window, in units of psia. Close the canister valve when the pressure reaches 30psia. There is a relief valve on the diluter that will open when the pressure reaches 35psia, so the canister will still be usable if the valve is not closed in time.

10.2.2.2 When analysis of additional (extra) compounds are requested which are not in the purchased stock cylinders, the following preparation instructions should be used. In addition, the internal standard / surrogate standard may also be prepared in this manner (Sections 10.2.2.2.1 – 10.2.2.2.2) as mentioned in Section 10.2.1.

10.2.2.2.1 *Equi-mass "soup"* (contains compounds in equal mass amounts) or *cocktail* prepared from the neat compounds for a large number of components. If additional SIM compounds are requested, the same cocktail may be used.

Cocktail Preparation:

Step 1: This cocktail is prepared by combining 25mg of each neat compound into a small glass vial. Use a microliter syringe to transfer each compound, cleaning with solvents in between. Put the vial in the freezer between aliquots to minimize volatilization. Take the density of each compound into account to determine the actual amount of each compound to spike into the cocktail by using the following equation.

$$S = \frac{A}{D} \qquad \text{(Equation 4)}$$

Where:

S Actual spike amount (µL)
A Desired amount for each compound (mg)
D Density (mg/µL); refer to Table 2 for the density

Example: The actual volume of acrolein to add to the cocktail is calculated by the following.

$$S(Acrolein) = \frac{25mg}{\left(0.840\frac{mg}{\mu l}\right)} = 29.8\mu L$$

Step 2: The concentration of each compound in the cocktail is determined by the following equation.

$$C = \frac{A}{V}\left(1000\frac{\mu g}{mg}\right) \qquad \text{(Equation 5)}$$

Where:

C Concentration of cocktail (µg/µL)
A Amount of each compound (mg)
V Final volume of cocktail (total spike volumes of each compound) (µL)

Example:

$$C = \frac{25mg}{631.8\mu L}\left(1000\frac{\mu g}{mg}\right) = 39.569\mu g/\mu L$$

10.2.2.2.2 *An intermediate standard* is prepared from neat compounds by spiking individual compounds into a glass static dilution bottle (SDB) as described in Section 10.2.1.1 or spiking an aliquot of a cocktail into the SDB. The spike amount of a cocktail is determined by using the following equation.

$$S = \frac{C_1 V}{C_2} \qquad \text{(Equation 6)}$$

Where:

S Spike amount required in order to obtain the desired concentration (µL)
C_1 Desired concentration of SDB (µg/mL)
C_2 Concentration of cocktail (µg/µL)
V Volume of SDB (L)

Example: Determine the spike amount of the cocktail required to achieve the desired intermediate standard concentration.

$$S = \frac{\left(1\frac{\mu g}{ml}\right)(2010ml)}{27.81\frac{\mu g}{\mu L}} = 72.28\mu L$$

10.2.2.2.3 *Intermediate Standard Preparation (Gaseous Compounds*) As an alternative to the glass SDB method, if the extra compounds needed to be analyzed are gases at room temperature, use a gastight syringe to prepare an intermediate standard in a 1L Tedlar bag filled with humidified zero-grade air. Use the molecular weight of the compound to calculate the microliter amount to be spiked into the bag to achieve desired concentration. The spike amount is determined by using the following equation.

$$S = \frac{C * V * 24.46}{M * \left(1000\frac{ng}{\mu l}\right)}$$

S	Spike amount required in order to obtain the desired concentration (µl)
C	Desired concentration (ng/L)
V	Volume of the Tedlar Bag (1L)
M	Molecular Weight of the compound
24.46	Molar Volume of gas at 25°C, 1atm

Example:

Make a 100,000ng/L intermediate standard of Chloro-difluoromethane (Freon22) in a Tedlar Bag, where M=86

$$S = \frac{100{,}000\frac{ng}{L} * 1L * 24.46}{86 * \left(1000\frac{ng}{\mu l}\right)} = 28.44\mu l$$

10.2.2.2.4 *The Working standard* for extra compounds is prepared in a Summa canister by spiking an aliquot of the intermediate standard (glass SDB or Tedlar bag) using a heated gastight syringe. The preparation of these standards shall follow the instructions detailed in Section 10.2.1.2. The concentrations for working standards are usually 20 and 200ng/L, however different concentrations can be chosen which work best for a particular project.

10.2.3 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) Prepare a secondary source standard (either a different manufacturer or different lot from the same manufacturer as the initial calibration standard) using the same procedures as the primary source. The ICV/LCS working standard should

contain each target analyte present in the calibration working standard. Prepare the ICV/LCS working standard at a concentration of 200ng/L. Differing injection volumes account for the allowed concentrations listed in Table 4 for SCAN and 4A for SIM. The preparation of this standard shall follow the instructions detailed in Section 10.2.2, using the certified second-source standard cylinder.

10.2.4 Continuing Calibration Verification (CCV) Standard The CCV is the same as the initial calibration working standards detailed in Section 10.2.2.

10.2.5 Screening Standards Recommended procedure: Prepare a 0.5ug/mL and/or a 3.0ug/mL concentration standard so that the GC may be calibrated utilizing a few levels (may include approximately 0.5ng, 150ng and 600ng). However, other concentrations can be prepared depending on the desired range.

Any of the desired standard concentrations (primary and secondary) may change as long as the equations and the appropriate densities remain the same.

10.3 Storage and Expiration Dates

All standards that are to be stored in a freezer shall be stored at ≤-10˚C for DoD projects.

- Neat Stock Liquids are stored at < -10˚C (-10˚C to -20˚C) as specified by the manufacturer or for a period of five years.
- Equi-Mass Primary Stock Standard is a cocktail or soup of neat compounds (containing compounds in equal mass amounts) used to in preparing intermediate gas phase standards and shall be stored in the freezer at < -10˚C (-10˚C to -20˚C) for up to six months. This is assuming that the soup is sealed with a septum-containing screw cap or Mininert™ valve. The selection of the compounds for the soup should be performed in accordance with the guidelines in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Purchased Stock Standards Cylinders must be stored at laboratory temperature for a period of 2 years or as specified by the manufacturer before vendor re-certification or purchase of new standards.
- Intermediate Calibration Standards prepared by static dilution must be stored in an oven at a temperature of approximately 60°C to ensure analyte vaporization. Every time a standard is prepared from the static dilution bottle (SDB), the concentration changes. To increase the useful lifetime of an SDB standard, remove volumes of 25mL or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, an SDB intermediate standard is stable for approximately two months as long as new working standards made from this standard continue to meet acceptance criteria. These bottles must be in the oven for a minimum of one hour prior to use in preparing working standards. The guidelines for the storage and expiration date for the intermediate calibration standards are stated in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Prepared Stock / Intermediate Calibration Standards prepared in Summa canisters (1000ng/L) may be stored at laboratory conditions for up to three months in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

- Calibration or Working Calibration Standards prepared in canisters may be stored at laboratory conditions for one month in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

11) Method Calibration

11.1 Initial Calibration

The initial calibration is performed to determine instrument sensitivity and the linearity of the GC/MS response for the target compounds.

Initial calibration requirements are as follows:

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. An initial calibration must be performed at a minimum initially per instrument, annually thereafter or whenever the continuing calibration verification standard does not meet the acceptance criteria.
3. Highest concentration, together with the lowest concentration, defines the calibration range.
4. The method reporting limit for any reported analyte must be at >/= the lowest calibration point.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from lowest to highest concentration.
8. All ICAL analyses must be completed within the 24-hour tune window.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. One of the calibration points from the initial calibration curve must be at the same concentration as the continuing calibration verification standard.
11. The upper end of the calibration range must not exhibit any peak saturation for any analyte or the range must be lowered accordingly.
12. The initial calibration model must be linear calibration using average of response factors and cannot be changed for any reason.
13. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at or below the MRL (LOQ) and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
    - Points at the high end may be dropped, but doing so lowers the calibration range.
    - Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).
    - Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but

the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.

- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 24 hours).
- Once the ICAL has been used to calculate and report sample results it MUST not to be changed for any reason.
- It is recommended that if an analyte has a higher MRL than the lowest concentration analyzed that the low standard be automatically dropped from the curve (i.e., acetone MRL is 5, drop at least the 0.4ng point).

11.1.1 Calibration Points Analyze the calibration standards (analyze low to high) that span the monitoring range of interest of the samples. For SCAN, the range is typically 0.4ng-100ng on column; however, 0.08ng on column may be added if low level analyses are requested. For SIM, the range is 10pg on column to 50,000pg on column. The dynamic range is dependent on the sensitivity of a particular instrument as well as the required reporting limit for a given project and may be adjusted accordingly. Refer to Table 3 (SCAN) and Table 3A (SIM) for the concentrations of the compounds of interest in the initial calibration at each particular calibration concentration level.

Note: Refer to the EXCEL TO-15 Standard Concentration templates, located on the network at Q:\\TO15 Std. Concentrations\Std. Conc. Templates for both the SIM and SCAN templates. These templates must be utilized for the documentation of the standard canister concentration selection, final ICAL level concentrations and the determination of the correct injection volumes for the selected standard canister concentrations. If the primary or secondary stock standard cylinder concentrations are revised (upon re-certification or new purchases), the EXCEL spreadsheet templates, injection amounts and the ICAL concentrations in each instrument method must be adjusted accordingly. Other templates may be employed as long as they are validated and provide at least the same information.

SCAN

1. Determine if the lower end of the calibration range is to be 0.08ng or 0.4ng on column. If the low end is 0.08ng, then the 1ng/L standard must be utilized.
2. Determine if the 1ng/L or 20ng/L standard canister is to be used for the 0.4ng on column point.
3. Follow the instructions in the spreadsheet and save the file under the correct instrument folder and the initial calibration method identification.
4. Print the final ICAL concentration sheets and place into the corresponding ICAL folder

11.1.2 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not met and at least annually. The following procedure must be followed when updating an initial calibration method.

1. Open the most recent method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID must be the date files were analyzed.

3. Quantitate midpoint standard and check retention times and integrations. Update retention times if necessary using QEdit or Easy ID (Tools → Easy ID). Requant if any changes are made and verify all peaks are identified correctly. Print.
   a. While midpoint standard is loaded update reference spectra (Continuing Calibration → Update Reference Spectra).
   b. With midpoint standard loaded update qualifier ion ratios and retention times (Initial Calibration → Update Levels → Select Update Level and then select Retention Times (Replace) and Replace Qualifier Ion Relative Responses).
   c. If necessary adjust integration parameters prior to processing remaining ICAL points.
4. Quantitate remaining ICAL standards. Review each peak for retention time, integration, and print. Review low level standards for acceptable signal to noise ratios and high level standards for saturation.
5. All responses must be cleared from ICAL before updating (Initial Calibration → Clear All Calibration Responses).
6. Update responses for each standard level (Initial Calibration → Update Levels) or (Initial Calibration → Quick Levels Update). If Quick Levels Update is used do not requant datafiles.
7. Save method.
8. Check Response Factor Report and evaluate whether any points should be dropped following the criteria outlined in this SOP.
9. Save method if any changes are made.
10. Verify calibration files listed on Response Factor Report are correct.
11. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report.

11.1.3 Analytical Window If time remains in the tune window after meeting the acceptance criteria for the initial calibration, samples may be analyzed according to the procedure described in this document (see Section 12.3.2). If time does not remain in the analytical window, a new sequence shall commence with the analysis of the instrument performance check compound (BFB) and the continuing calibration verification standard.

11.1.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 12.4. Use the standard prepared in accordance with Section 10.2.2 of this SOP. Attach the calibration standard and internal standard/surrogate canisters to the designated inlets on the preconcentrator and open the canister valves. Analyzing different volume aliquots of the calibration standards produces differing concentrations.

Analyte responses (target ion areas) are tabulated and recorded using the Enviroquant program. Quantitation ions for the target compounds are shown in Table 2 and 2A and the primary ion should be used unless interferences are present, in which case the secondary ion may be used, but the reason documented in the initial calibration file and all subsequent quantitations utilizing that ICAL must be performed using the same ion selections. Refer to Section 15.2 for the required calculations and Section 16.4 for the acceptance criteria.

11.1.4.1 Additional Requirements The procedure for performing and generating a new initial calibration method must follow a few additional requirements.

1. If any analyte lacks the appropriate sensitivity (3 to 1 signal to noise ratio) at the low end of the calibration range, this point must be dropped from the curve and the MRL/LOQ raised accordingly.
2. No detector saturation may occur for any compound; the upper calibration level must produce no saturated peaks. Exhibited by:
   - The flattening of the response for the higher concentration standards as shown on the plot;
   - The presence of a reverse tail or rise on the front part of the peak;
   - The observed actual percent ratio of the secondary ion presence is lower than the expected percent ratio; or
   - The presence of a flat topped peak and again by the decline or saturation of the secondary ion compared with the expected % recovery.

11.1.4.2 LOQ Establishment, Verification and Acceptance Criteria

1. The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
2. The LOQ for each analyte must be ≥ the analyte's LOD.
3. Initially a passing demonstration of precision and bias must be performed at the LOQ.
4. Run CCV 2 times at LOQ and:
   a. Generate a duplicate report for precision using ±25% as the criteria.
   b. Check the %Rec using laboratory generated control limits.
   c. Check the signal to noise ratio (S/N) using the software. The S/N ratio must be at least 3:1 for each analyte.
   d. All ion abundances must be acceptable per the requirements set forth in this document.
5. If any compounds fail, verify at a higher level and notify reporting. Also, make a note in the ICAL documentation.
6. Turn in all LOQ verification data (quant reports and software reports/checks) to QA (regardless of pass/fail).
7. Verify the LOQ on each instrument quarterly.

11.1.5 Initial Calibration Review Analyst's calculation and assessment along with a peer review of all ICAL data and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples. In the case where samples are placed on the autosampler and allowed to run overnight, the sample results may only be reported if the ICAL is reviewed and found to be acceptable. The ICAL checklist in Attachment 2 must be used to document the review and approval process.

Perform a review of specific aspects of the calibration which might compromise data quality such as inappropriate extension of the calibration range with detector saturation and/or a lack of sensitivity for any analyte. Analyte concentrations which do not meet the signal to noise ratio or exhibit saturation are not to be reported and must be eliminated from the initial calibration. These instances should be followed by a short explanation regarding the reason for the omission.

11.1.6 Initial Calibration File An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- BFB tune analysis report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and % recovery report.
- TO-15 Standard Concentration Spreadsheet (exact ICAL level concentrations and ICV concentrations)
- Any manual integration documentation

11.2 Initial Calibration Verification Standard

Verify the initial calibration by analyzing an initial calibration verification standard (ICV). This standard shall be obtained or prepared from materials acquired from a different manufacturer or lot from that of the initial calibration and prepared according to Section 10.2.3.

Analyze 50ng or less (refer to Table 4 for the secondary source standard concentrations) of the ICV standard depending on the dynamic range of a given instrument and refer to Section 15.4 for the required calculations.

12) Sample Preparation/Analysis

12.1 Sample Preparation

The pressure/vacuum is checked and the canister pressurized upon receipt by the laboratory, as needed. When necessary, canisters shall be pressurized with humidified zero grade air. However, if the samples are to be analyzed in accordance with EPA Method 3C then the samples must be pressurized with UHP Helium (refer to Section 12.9 for additional information). The client must be made aware of this in advance and given the option of either submitting two canisters for analysis or receiving a report with qualified results (TO-15 Modified).

Depending on the size of the canister and location of sampling and as specified in the SOP below, samples may be pressurized to approximately 1.0psig to 3.5psig. Additional information may be found in the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters*. Initial and final pressures are recorded in LIMS and should be repeated on the back of the sample tag. The dilution factor created by filling the sample canister is calculated using equation number 12 in Section 15.7.

12.2 Screening

The analyst must screen a sample or subset of samples if the source is of unknown origin. Typically, if the source is known to be indoor or ambient outdoor air, no screening is necessary. However, if screening is required make sure that the instrument is calibrated. A single point calibration is sufficient; however, the instrument may be calibrated utilizing a two point calibration. The ICAL points are recommended to be at approximately 0.5ng, 150ng and/or 600ng spanning the desired dynamic range. Refer to Section 10.2.5 for additional information.

Inject a 1mL or smaller aliquot of each sample into a GC/flame ionization detector (FID) system that has been calibrated with a standard containing a subset of the target analytes. This subset represents the most commonly found compounds in air samples, such as acetone, trichloroethylene, and toluene. Use the results to determine the maximum volume of sample to be analyzed by TO-15 by utilizing the following equation. Dilutions may be prepared as necessary according to Section 12.9.1.

$$I = \frac{C}{H}$$

Where:

I Injection volume (mL)
C Maximum calibration level (ng on column)
H Compound screening concentration (ng/mL)

Example: Select the compound with the highest concentration (toluene = 1.0ng/mL). If the upper calibration level is 100ng on column, then the following calculation determines the maximum injection volume to analyze.

$$\frac{100ng}{1.0ng/mL} = 100mL \text{ maximum injection volume}$$

12.3 Analytical Sequence and Data System Setup

12.3.1 Data System For the Tekmar AUTOCAN, fill in the sequence log of the Teklink program with the appropriate information. Refer to the Section 12.4.1 for the operating parameters.

For HP Chemstation, load the appropriate acquisition method for the GC/MS in the top window of the Chemstation program. Suggested GC/MS operating parameters are given in Section 12.4.2.

12.3.2 Analytical Sequence The analytical sequence must be completed for the analysis of ≤20 (19 samples including dilutions with one laboratory duplicate) field samples. A method blank (MB) shall be run to monitor for laboratory introduced contamination. There must be at a minimum a laboratory duplicate (LD) analyzed in each batch to access batch precision. The following generalized analytical sequence is to be followed:

Analytical Sequence Guideline

With Calibration	Tune Check[1] Calibration Standards (5 Standards Minimum) ICV Standard[2] (Acts as the ICV and LCS) QC Canister Checks[6] MB[7] Sample(s) – 1-19 Laboratory Duplicate[4]
With Continuing	Tune Check[1] CCV Standard[5] QC Canister Checks[6] MB[7] LCS[3] MRL Check Standard[8] Sample(s) – 1-19 Laboratory Duplicate[4]

[1] The instrument performance check solution must be analyzed initially and once per 24 hour (or as specified by the project) time period (sequence / tune window) of operation. All analyses for a sequence must be initiated (injected) prior to the expiration of the tune window.

[2] In this scenario, the ICV may also be evaluated as the LCS (differing acceptance criteria).

[3] An LCS shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard analyzed at the lower end of the calibration curve (below the midpoint).

[4] A laboratory duplicate must be analyzed at a rate of 1 per 20 or fewer samples. The duplicate must be rotated among clients, whenever possible. Also, a duplicate laboratory control sample may be analyzed to assess precision to meet project requirements or due to sample matrix effects.

[5] A CCV must be analyzed at the beginning of every analytical sequence.

[6] Any number of QC check canisters may be analyzed in the sequence to determine a canister cleaning batch or batches acceptability.

[7] Any of the QC Check Canisters may serve as the method blank as long as the minimum requirements detailed in this document are met. A method blank shall be analyzed at a rate of 1 in 20 or fewer samples.

[8] A MRL check standard may be analyzed with each batch of 20 or fewer samples (when an initial calibration is not analyzed within the same batch). Additional information is included in Section 12.15.

Note: Client project batch specifications may require certain modifications to the analytical sequence; however, a batch may not be more lenient than that which is specified in this document.

12.4 Conditions

12.4.1 Sample Collection Conditions The suggested settings and system parameters are as follows:

Adsorbent Trap

Set Point:	35˚
Sample Volume:	up to 1L
Dry Purge:	300mL
Sampling Rate:	100mL/min (utilize for a sample injection volume of >100mL); 40mL/min (utilize for a sample injection volume of 25-100mL)
Desorb Temp.:	200˚C to 230˚C
Desorb Flow Rate:	8-10mL/min He
Desorb Time:	3.0 minutes

Refocusing Trap

Temperature:	-180˚C
Injection Temp.:	160˚C
Injection Time:	1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature:	265°C

Initial Bakeout: 2 hours or until clean blank is obtained
After each run: 5-8 minutes

Sample Run Time

Each analytical run is approximately 20 minutes long; the total cycle time is about 30 minutes between injections.

12.4.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

Item	Condition
Carrier Gas	Helium
Flow Rate	1.0-1.6mL/minute
Temperature Program	Initial Temperature: ~20˚C Initial Hold Temperature: 3 minutes Ramp Rate: 5˚C/min to 80˚C 2nd Ramp: 10˚C/min to 160˚C 3rd Ramp: 20˚C/min to 240˚C for 5 min hold
Detector B (MSD *Interface)*	260˚C
Electron Energy	70 Volts (nominal)
Mass Range (Scan mode)	34 to 280 amu
Mass Range (SIM mode)	Scan masses corresponding to the target analytes
Scan Time	To give at least 10 scans per peak, not to exceed 1 second per scan.

Note: The instrument may be operated in Selective Ion Monitoring (SIM) mode if requested by the client.

12.5 Instrument Performance Check

Since the BFB tuning compound is included in the internal standard and surrogate standard canister and an autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

The mass spectrum of BFB must be acquired in the following manner.

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.
- All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.
- The ion abundance criteria must not be changed from the requirement stated in this document (TO-15 or TO-14A, as requested).

All subsequent standards, samples and QC samples associated with a BFB analysis must use identical instrument conditions.

12.6 Continuing Calibration Verification Standard

Verify the calibration each working day, where necessary (e.g., an ICAL was not analyzed or the tune window has closed) by analyzing a continuing calibration verification (CCV) standard from the initial calibration standard canister. The concentration of the calibration verification may be varied between the low calibration standard and the midpoint of the calibration range; however, the concentration must be at one of the levels analyzed in the initial calibration. Refer to Table 3 for the standard concentrations. Refer to Section 15.3 for the required calculations.

12.7 Canister Quality Control Check and Method Blank

The method blank must be a sample of a matrix similar to the batch of associated samples that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure, and in which no target or interferences are present at concentrations that impact the analytical results for sample analyses. Prepare a canister that has not left the building by pressuring with humidified zero air. Analyze an aliquot of one liter along with the same volume of internal standard and surrogate as standards and samples. Additionally, a blank must be analyzed whenever a high concentration sample is encountered and carryover is suspected.

A Quality Control (QC) check canister pressurized with humidified zero air may serve as a method blank as long as the analyte concentration requirements stated in the canister quality control check section (Sections 16.7 and 16.8) and other requirements (refer to Section 16.12 for internal standard requirements) are met. Assuming continuing failure, another QC canister or a new canister must be prepared and analyzed in order to verify that no system contamination exists. For tracking purposes the unique laboratory barcode given to a canister shall be the information included in the sample analysis identification.

12.7.1 Sampling Systems Section 7.1 and 8.4 of Method TO-15 describe the setup and certification procedure for a specific sampling apparatus that has been used by the EPA for several of its large air monitoring programs. These systems are rarely used for the types of projects that make up the bulk of the laboratory's work. The vast majority of samples analyzed by the laboratory are taken into Summa canisters either as grab samples or using a simple time integrated sampling device (flow controller), as in Section 8.2.1 of the method, so these procedures are not part of the typical protocol for providing sampling materials to clients. The laboratory has developed an SOP for the cleaning and certification of the materials it provides its clients for obtaining air samples to be analyzed by method TO-15. Refer to the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters* for additional information.

It is this laboratory's interpretation that the sampler system certification procedure described in Section 8.4.4 of the TO-15 method applies to the specific sampling apparatus described in the method and not to the sampling procedures used by our clients. The laboratory does not maintain a dynamic calibration manifold or canister sampler apparatus as described in the method and thus performance of the relative accuracy certification procedure described in section 8.4.4 is not possible.

12.8 Laboratory Control Sample

The laboratory control sample is a sample matrix, which is free from the analytes of interest and spiked with a standard containing known amounts of analytes. The

laboratory control sample is an injection of the initial calibration verification standard. Inject the LCS (ICV) at concentrations below the midpoint of the calibration curve. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

12.9 Sample Analysis

Prior to analysis, all sample containers (canisters and bags) should be at temperature equilibrium with the laboratory.

- Attach sample canisters to Tekmar AUTOCan using a 9/16" wrench. Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments). Tedlar bags can be connected using soft silicone tubing or a 3/16" fitting with a reusable ferrule.
- Before opening the valve, check for leaking fittings by running the leak check program in the Teklink software. Quick connect fittings must be leak checked before connecting the sample container.
- If system is leak tight, open the canister valves and start the automated preconcentration procedure. Make sure the Chemstation data acquisition software has been readied.
- Maintain the trap at an elevated temperature until the beginning of the next analysis.

Check all target compounds using the QEdit routine in Enviroquant, making sure all extracted ion chromatogram peaks are integrated properly (see Section 12.13).

Note 1: The secondary ion quantitation is only allowed if there is sample matrix interference with the primary ion. If the secondary ion quantitation is performed, document the reasons in the instrument run logbook and/or on the quantitation report (initial and date any notation).

Note 2: Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fitting higher than 80ºC.

SCAN Mode - The instrument is normally operated in the SCAN mode, where the following procedure may be followed.

- Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic range from 34 to 270 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning allows identification of unknown compounds in the sample through searching of library spectra. See operating conditions in Section 12.4.
- Generate a quantitation report for each run.
- If reporting Tentatively Identified Compounds (TICs), refer to Section 12.9.2 for identification criteria.

SIM Mode - When the client requests SIM mode, select SIM instead of SCAN mode and identify a minimum of two ions per analyte of interest. Also, a minimum of two ions for each internal standard and surrogate compound should be selected.

Helium Pressurization – If a canister is pressurized with helium, a correction factor is applied to sample volumes extracted from the canister via auto sampler. This is due to the difference in thermal properties between helium and air. A correction factor worksheet has been generated to determine the exact volume taken from a canister and may be found at J:\\A-GCMS\Helium Pressurization. Save file, print the sheet and include with the data. Refer to the instruction page in the template for all of the instructions and calculations including backfilled canisters.

AutoCAN Leak Checks – Canisters should be put on at least two different AutoCAN positions to confirm a "leak". In addition, the valve threads should be inspected for defects which may prevent a good seal with the AutoCAN. Once a canister has "failed" the leak check it must be tagged, an NCAR initiated, and the PM notified. Regardless of what the client or PM specifies as the fate of the sample, the canister must be put on maintenance hold to complete a full 24-hour leak check. A yellow sheet is to be completed in addition to, but not in lieu of an NCAR. This is a fixed QA procedure with no allowance for deviation.

12.9.1 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot should be analyzed. The dynamic range of volume aliquots for the automatic cryogenic concentrator is 20cc to 1L. If a volume smaller than 20cc is to be analyzed, a dilution should be made in a Tedlar bag, or the sample directly injected using a gastight syringe. Guidance in performing dilutions and exceptions to this requirement are given below.

- Refer to Section 12.4.1 (Adsorbent Trap Sampling Rate) for the required sampling rate if less than 100mL is to be analyzed.
- Use results of the original analysis to determine the approximate dilution factor required and get the largest analyte peak within the initial calibration range.
- The dilution factor must be documented (and included in the final report) and chosen in such a way as to keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument.

Tedlar bag dilution:

- Make a dilution by filling a Tedlar bag with 1.0 liter of humidified zero air using a one-liter gas syringe.
- Calculate the volume of balance gas needed to obtain the required dilution.
- Remove the difference in the balance gas using a syringe.
- Add the calculated sample amount using a gastight syringe.

Direct injection:

- Make a direct injection by attaching a clean, humidified zero air filled Summa canister to the preconcentrator autosampler using 1/4" stainless steel or teflon tubing with a "tee" septum port. This canister should be the same canister that may be used as the method blank.
- Inject the sample through the septum while the preconcentrator withdraws a 200cc aliquot from the canister.

12.9.2 Tentatively Identified Compounds When requested, a mass spectral library search may be made for the purpose of tentatively identifying sample components not associated with the calibration standards. The necessity to perform this type of identification will be determined by the purpose of the

analyses being conducted. Data system mass spectral library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Certain programs may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. The following guidelines are used for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. For example, for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30 and 70%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- The concentration of the tentatively identified compound is estimated by assuming a response factor of 1.0 and comparing the response of the tentatively identified compound to the response of the nearest internal standard.
- If non-target analytes are not Q-deleted from the quant report, the analyst must evaluate whether these compounds should be reported as TICS.

12.10 Duplicate

A duplicate must be analyzed to assess laboratory precision and samples selected for duplicate analysis shall be rotated among client samples, where applicable. Some projects or sample matrix issues may require the analysis of a duplicate laboratory control sample (DLCS).

12.11 Internal Standard (IS)

The concentration of internal standard added to each standard, field sample and QC sample must be consistent from that of each current ICAL standard.

12.12 Surrogates

Internal standards/surrogates must be added at the same volume for every standard, sample and QC sample. Surrogate compound recoveries are requested by a number of clients, but are more appropriately used as system monitoring compounds. This is due to the fact that the compounds are introduced directly into the analytical system and not into the canisters or bags. It is for this reason that they are not considered to be true surrogates and a fixed window is applied. Additionally, surrogates are not included in the ICAL because they are not required by the method and are only system monitoring compounds.

12.13 Manual Integration and Q Deletion

A list of abbreviations (codes) that may be used to give a reason for performing either of these procedures are listed in the *SOP for Data Review and Reporting*.

12.13.1 Manual Integration The integration for each peak must be legally defensible and shall be checked to ensure that it has been integrated properly and consistently between samples, standards and QC samples. All peak reviews and manual integrations must follow the requirements specified in the *SOP for Manual Integration Policy* and the *SOP for Laboratory Ethics and Data Integrity.* The requirements in the above stated procedure include when manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., chromatograms showing both the integration prior to any manual integrations and those depicting the corresponding manually integrated peaks), and notation of rationale, date, and initials of person performing the manual integration operation. In addition, manual integrations must be reviewed and approved by a second reviewer and the manual integrations maintained in the appropriate job file.

Reporting Requirements Certain project requirements including samples which are submitted under the Department of Defense (DoD) QSM require that the case narrative include an identification of samples and analytes for which manual integration is required. Refer to project requirements to determine if this is necessary.

12.13.2 Q Deletion Q deleting may be performed to either delete a false positive or delete non-target compounds.

12.14 Detection Limits and Limits of Detection

The MDL study shall be performed annually for all target analytes on each instrument (with identical configurations) for which this method is performed. The MDL shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The detection limit shall be used to determine the LOD for each analyte.

Once determined on each instrument, the highest LOD (for each analyte from all instrument determinations) shall be used as the uniform LOD. However, if a lower detection limit is reported, then the samples must have been run on that specific instrument on which the lower LOD was determined.

12.14.1 Performance and Acceptance Criteria

1. The MDL must be <0.5ppbV for each analyte (Method 11.11.1).
2. Perform Limit of Detection (LOD) verification on all instruments (performing this method) immediately following the MDL study. Spike the LOD at 2-4x the MDL; the spike level establishes the LOD.
3. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).
   - It is specific to each combination of analyte, matrix, method and instrument configuration.
   - The LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.
4. If the LOD verification fails (per #3), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.
5. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

12.15 Method Reporting Limit Check Standard

It is recommended to analyze a MRL check standard at the current MRL or required MRL for the batch (per client requirements) of twenty or fewer samples if the CCV fails low for any target compound. A MRL check standard may also be required per client specifications.

This check standard can also serve as the LOQ verification if it meets the specific requirements listed in Section 11.1.4.2. Apply the requirements and retain all documentation accordingly. Refer to Attachment 4 for Minnesota specified MRL check standard criteria.

12.16 Method Modifications

Method modifications are not allowed under NELAC\TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 12.9.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. There are multiple quantitation methods, which are subsets of the compound list in Table 2. Therefore, files will be named with an eight-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files are saved in a unique sub-directory on the server.

14.2 Sufficient raw data records must be retained on file of all laboratory analyses described in this document including passing QC canister checks, tune checks, instrument calibrations, verifications, sample analyses and dilutions, QC checks, and method detection limit studies. The information that is required includes: analysis/calibration date and time, test method, instrument, sample identification, analyte identification, analyst's initials, concentrations and responses, as well as standards used for the analysis and calibrations, all manual calculations including sample dilutions and manual integrations to permit reconstruction of analyses. Information entered and reported on the quantitation report and instrument run log must be complete and accurate. All data shall be obtained following defensible and ethical practices in accordance with the most recent Quality Assurance Manual and the *SOP for Laboratory Ethics and Data Integrity*.

Note: All data records must explicitly connect data to the initial instrument calibration. This includes all samples, continuing calibrations and QC samples.

14.3 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time (if the holding time is 72 hours) of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings and balance gas if pressurized with helium), standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 This method has specific requirements including the use of canisters; any modification must be reported accordingly. All reports that fall under the laboratory's certificate of approval (in accordance with NELAC/TNI standards) must include a statement(s) clarifying any deviations from the scope of this certification. Refer to Section 15.10 for additional information and specific items, which require this clarification.

15.2 Initial Calibration

Tabulate each of the following:

15.2.1 Equation Number 1 - **Relative Response Factor (RRF):**

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$ where:

A_x is the area response of the analyte quantitation ion.
A_{is} is the area response of the corresponding internal standard quantitation ion.
C_{is} Internal standard concentration, ng.
C_x Analyte concentration, ng.

Note: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC samples is the same from run to run.

15.2.2 Equation Number 2 - **Average (or Mean) RRF:**

$$\overline{RRF} = \frac{\sum_{i=1}^{N} RRF_i}{N}$$ where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

N is the number of calibration concentration levels.

15.2.3 Equation Number 3 - **Standard Deviation, SD:**

SD = $\sqrt{\sum_{i=1}^{N} \frac{\left(RRF_i - \overline{RRF}\right)^2}{N-1}}$ where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

$\overline{RRF}$ Average (or Mean) RRF of all concentration levels in the initial calibration curve.

N total number of calibration concentration levels

15.2.4 Equation Number 4 - **Percent Relative Standard Deviation, %RSD:**

%RSD = $\frac{SD}{\overline{RRF}}(100)$ where:

SD Standard Deviation calculated in equation number 3

$\overline{RRF}$ Average or Mean RRF

15.2.5 Equation Number 5 - **Relative Retention Time (RRT):**

$$\mathrm{RRT} = \frac{\mathrm{RT_C}}{\mathrm{RT_{is}}}$$ where:

RT_C Retention time of the target compound, seconds.

RT_{is} Retention time of the internal standard, seconds.

15.2.6 Equation Number 6 - **Mean Relative Retention Time ($\overline{RRT}$):**

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT_i}{n}$$ where:

$\overline{RRT}$ Mean relative retention time (seconds) for the target compound for all initial calibration levels.

RRT_i Relative retention time for the target compound in level i.

n Number of calibration levels

15.2.7 Equation Number 7 - **Mean Area Response ($\overline{Y}$):**

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$ where:

Y_i Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

n number of calibration concentration levels

15.2.8 Equation Number 8 - **Mean Retention Times ($\overline{RT}$):**

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$ where:

$\overline{RT}$ Mean retention time, seconds
RT_i Retention time for the internal standard for each initial calibration standard, seconds.
n number of initial calibration levels

15.3 Continuing Calibration Verification

- Calculate the (RRF) of each target compound using equation number 1.

15.3.1 Equation Number 9 - **Percent Difference, %D:**

$$\%D = \frac{RRFx - \overline{RRF}}{\overline{RRF}}(100)$$ where, for any given analyte:

RRF_x is the RRF from the CCV being evaluated.
$\overline{RRF}$ is the mean RRF from the current calibration curve.

15.4 Percent Recovery – ICV, LCS, Surrogates, MRL Check Standard

15.4.1 Equation Number 10 - **Percent Recovery (%R):**

%R = X/TV x 100

where
X = Concentration of the analyte recovered
TV = True value of amount spiked

15.5 Duplicate Analysis

15.5.1 Equation Number 11 - **Relative Percent Difference (RPD):**

$$\frac{x_1 - x_2}{\overline{x}}(100)$$ where:

x_1 First measurement value
x_2 Second measurement value
$\overline{x}$ Average of the two values

15.6 Internal Standards (IS)

- Calculate the mean area response $\overline{Y}$ for each internal standard using equation number 7.
- Calculate the mean of the retention times for each internal standard using equation number 8.

15.7 Pressure Dilution Factor (PDF)

15.7.1 Equation Number 12 - **PDF**, for samples collected in Summa canisters:

$$PDF = \frac{P_{atm} + P_f}{P_{atm} + P_i}$$ where:

P_{atm} is the ambient atmospheric pressure, 14.7 psi at sea level.
P_f is the final sample canister pressure, in psig.
P_i is the initial sample canister pressure, in psig. This will most often be a negative value (sub-ambient initial pressure).

15.8 Results

If a canister has been pressurized with Helium and the Tekmar AutoCan was utilized, refer to Section 12.9.

15.8.1 Equation Number 13 - For calculating analyte concentrations in a sample, the starting point is the nanogram amount generated by the HP Enviroquant software, which appears on the quantitation report.

$$ng_x = \frac{A_x ng_{is}}{A_{is}\overline{RRF}}$$ where:

ng_x is the nanogram amount of analyte *x*.
A_x is the area response of the analyte's quantitation ion.
A_{is} is the area response of the corresponding internal standard's quantitation ion.
ng_{is} is the internal standard amount, in nanograms.
$\overline{RRF}$ is the average or mean RRFs

15.8.2 Equation Number 14 - The final analyte concentration, C_x, in units of micrograms per cubic meter (µg/m³), is then calculated from the following:

$$C_x = \left(\frac{ng_x PDF}{V}\right)\left(\frac{1\mu g}{1000ng}\right)\left(\frac{1000l}{1m^3}\right)$$ where:

V is the sample volume analyzed, in liters.
PDF is the sample canister pressure dilution factor.

15.8.3 Equation Number 15 - To convert to units of parts per billion volume (ppbv):

$$ppbv = \frac{\mu g/m^3}{MW} x 24.46 \qquad \mu g/m^3 = \frac{ppbv}{24.46} xMW$$ where:

MW is the molecular weight (Table 2) of the analyte, in g/mole.
24.46 is the molar volume of an ideal gas at 298 K (25 °C) and 760 mmHg (1 atm), in liters per mole (l/mol).
C_x the final analyte concentration in micrograms per cubic meter.

15.8.4 Equation Number 16 – Helium Pressurization (Injection Amount)

Applicable to canisters pressurized with helium and injected utilizing the mass flow controller of the AutoCAN. For full instructions and calculations, refer to the 1st tab of the template located at: J:\A-GCMS\Helium Pressurization\MFC_GCF _backfill.

15.9 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated by analytical sequence following the Daily QC review checklist (Attachment 3). The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The Sample Review checklist (Attachment 3) is used to document sample review per service request and once completed, initialed and dated must be filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file organized by instrument and date. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.6.

15.10 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and information required by this laboratory's policy, NELAC\TNI standards, DoD Manual (applicable version, see reference section), client projects, and the TO-15 method including modifications, observances, data qualifiers, and certification information.

If the project requires that results be reported below the MRL (LOQ), but above the LOD all of the requirements specified for normal reporting apply (3:1 S/N ratio and ion abundance). This is regardless of the fact that the results will be qualified as estimated.

15.10.1 Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, must be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. All data qualifiers and flags should follow those listed in the most recent Quality Assurance Manual or as defined in any client requirements.

This form is necessary as a means for documentation. This form, among other information, will be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

15.10.2 NELAC\TNI Requirements

The following items do not comply with NELAC\TNI standard requirements and must be reported accordingly. A statement, however worded, must be

included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval.

- Reporting any compound which is not included in the second source standard (ICV or LCS) does not meet NELAC requirements.
- In addition, a report that contains a compound not included on the NELAC certificate of approval must also include the statement listed above.

15.10.2.1 Modifications

Method modifications are also not allowed under NELAC\TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 12.9.

15.10.3 Surrogates

Only report surrogates at the request of the client. If any surrogate is out of control, all samples results (with surrogates requested) associated with the surrogate must be reported with the appropriate data qualifier.

15.10.4 DoD Requirements

Report results with the appropriate data qualifiers, if samples cannot be reanalyzed for any reason. In addition and at a minimum, the following situations are to be noted in the case narrative: manual integrations, CCV out of control, and results exceeding the calibration range.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.2 Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate. Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV or any batch QC.

16.3 Instrument Performance Check

16.3.1 Acceptance Criteria

Refer to Tables 1 and 1A for the required ion abundance criteria.

16.3.2 Corrective Action Perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria. An acceptable tune is required for sample results to be calculated and reported.

16.4 Initial Calibration

16.4.1 Acceptance Criteria Refer to the following acceptance criteria for the initial calibration.

- The RRT for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound.
- The calculated %RSD for the RRF for each compound in the calibration standard must be less than 30% with at most two exceptions up to a limit of 40% (this may not be true for all projects).
- For each Internal Standard the area response (Y) at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range.
- The retention time shift for each of the internal standards at each calibration level must be within 20s of the mean retention time over the initial calibration range for each internal standard.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable ICV.

16.4.2 Corrective Action Follow the initial calibration requirements detailed in Section 11.1 for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards.

If the initial calibration results are outside the established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed, if reanalysis of the samples is not possible, data associated with an unacceptable initial calibration shall be reported as estimated with the appropriate data qualifiers.

16.5 Initial Calibration Verification Standard (ICV)

16.5.1 Acceptance Criteria The percent recovery for each compound in the ICV must be between 70%-130% for all analytes except vinyl acetate, which must be within 50-150%. Exceptions to this allowance for the vinyl acetate recovery are project specific requirements and any DoD type project, which shall adhere to the 70-130% requirement for all target compounds.

16.5.2 Corrective Action If the initial calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. Perform another initial calibration or if reanalysis is not possible, data associated with an unacceptable ICAL/ICV shall be reported as estimated with the appropriate data qualifiers.

16.6 Continuing Calibration Verification (CCV)

16.6.1 Acceptance Criteria All compounds must be evaluated prior to rounding. The percent difference for each target analyte must be within plus or minus 30% of the initial calibration average RRFs.

16.6.2 Corrective Action If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed; however, refer to 16.5.1 below.

16.6.2.1 Method Reporting Limit Check Standard

If the MRL check standard is unacceptable for any compound (sensitivity; ratio or %D), reanalyze at the same or higher level within the same batch and report data with the CCV flag and case narrative notes accordingly.

16.6.3 *DOD REQUIREMENT:* If a CCV fails, the laboratory must immediately analyze two additional consecutive CCVs (immediately is defined as within one hour).

- Both of these CCVs must meet acceptance criteria in order for samples to be reported without reanalysis
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

16.7 Canister Quality Control Check

The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included. If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual – DoD QSM, etc.).

16.7.1 Scan Analyses A canister is considered "clean" for normal SCAN analyses if the analysis shows <0.2ppbv of any target analyte (analyte exceptions listed in table below). If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual - DoD QSM, etc.).

Low Level SCAN Analyses For those analytes with a MRL of 0.1ug/m3, the QC criteria of <MRL is acceptable; otherwise, <0.2ppbV is required (analyte exceptions listed in table below).

SIM Analyses Results <MRL will be acceptable as this complies with the <0.2ppbV method requirement.

ANALYTE EXCEPTION LIST					
Compounds	ppbV	On Column (ng)	Compounds	ppbV	On Column (ng)
Target Analytes	0.2	0.50	Acrylonitrile	0.2	0.43
Chloromethane	0.2	0.41	Acetone	1.5	3.5
1,3-Butadiene	0.2	0.44	Ethanol	1.9	3.5
Acetonitrile	0.2	0.33	Vinyl acetate	0.99	3.5
Acrolein	0.65	1.5	1-Butanol	0.23	0.70
Isopropanol	0.28	0.70	Carbon Disulfide	1.1	3.5
2-Butanone	1.2	3.5			

Document the status of the check in LIMS and return the canister to the canister conditioning room. Additionally, if the check was found to be acceptable, the quantitation report must be kept on file for future reference

16.7.2 Tentatively Identified Compounds (TIC) If the batch of canisters are to be used for tentatively identified compounds (TIC) analysis, any non-target peaks present in the QC check canister analysis must be evaluated and determined to be less than the TIC reporting limit (10% of the internal standard). The concentration is estimated by assuming a RRF of 1.0 and comparing the response of the TIC to the response of the nearest internal standard.

16.8 Method Blank

16.8.1 Acceptance Criteria

- The concentration of a targeted analyte in the blank cannot be at or above the MRL, AND be greater than 1/10 of the amount measured in any associated sample. For any project that requires reported results less than the MRL, all associated measurements found in the MB should result in a qualifier; however, project requirements may differ and must be followed. Refer to DoD requirements listed below.
- The method blank should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.
- For DoD samples, the method blank will be considered to be contaminated if:

  1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
  2. The concentration of any common laboratory contaminant (acetone, ethanol, carbon disulfide, and methylene chloride) in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
  3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

16.8.2 Corrective Action If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results

are acceptable or prepare a canister per Section 12.7. If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). In all cases, the corrective action (reprocessing or data qualifying codes) must be documented. However, the specific corrective action depends on the type of project the blank is utilized for; therefore, refer (below) to the reporting/reprocessing requirements.

DEPARTMENT OF DEFENSE (DoD) QSM PROJECT: Any sample associated with a blank that fails the criteria shall be reprocessed in the same or subsequent analytical batch, except when the sample analysis resulted in a non-detect. If reanalysis is not performed, the results shall be reported with appropriate data qualifier.

OTHER PROJECT TYPE: Appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 18.4.

16.9 Laboratory Control Sample (LCS)

16.9.1 Acceptance Criteria Round all results to the nearest whole number prior to determining if the acceptance criteria have been met. The percent recoveries must be within the laboratory-generated limits and are referenced in the electronic TO-15 Method Manual. However, Arizona requires the percent recovery for each compound in the LCS to be 70%-130% (to match the ICV requirement). Therefore, the ICV exception for vinyl acetate stated in Section 16.5 requires the percent recovery for AZ samples to be 50-150%.

Note: Client project requirements, AFCEE and DoD requirements shall take precedence over the AZ requirement for AZ samples. Meaning if a sample is collected for a DoD project in AZ, DoD requirements specified in this document and the project specific QAPP (if supplied) are to be followed.

DoD Requirement: In the absence of client specified LCS reporting criteria, the LCS control limits outlined in the DoD QSM 5.0 Appendix C tables shall be used when reporting data for DoD projects.

16.9.2 Corrective Action If the LCS criteria are not met, determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and if the problem is with the injection re-analyze the LCS. DoD considers the same analyte exceeding the LCS control limits two out of three consecutive LCS to be indicative of non-random behavior; therefore, this trend should be monitored and the appropriate corrective action taken when it occurs.

16.10 Sample Results

16.10.1 Acceptance Criteria

- Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.

- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria described in this document.
- All target analyte peaks must be within the initial calibration range, diluted or reported with the appropriate data qualifier.

16.10.2 Corrective Action

- If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis as needed.
- If the area for any internal standard changes by more than ±40 percent between the sample and the most recent calibration, check for possible matrix interferences and re-analyze at a greater dilution. If the requirement is still not met and matrix interference is not detected the GC/MS system must be inspected for malfunction and maintenance made where necessary.
- When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

  To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.11 Laboratory Duplicate

16.11.1 Acceptance Criteria The relative percent difference must fall within ±25%. This RPD criterion also applies to duplicate laboratory control samples (DLCS).

16.11.2 Corrective Action If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.12 Internal Standards

16.12.1 Acceptance Criteria The following acceptance criteria must be applied to each run (except the ICAL – see Section 16.4).

- The area response for each internal standard in the blank must be within ±40 percent of the area response for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).
- The retention time for each internal standard must be within ±0.33 minutes of the retention time for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

16.12.2 Corrective Action

- Internal Standard Responses If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If

the problem is corrected with the lower volume analysis, report those results.

- Internal Standard Retention Times If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis where required.

16.13 Surrogates

16.13.1 Acceptance Criteria Since the matrix precludes the use of true surrogates and there is no established method criterion, acceptable surrogate recoveries are based on a fixed window of 70 - 130%. This is the typical requirement from clients. Additionally, these limits are referenced in SW-846 for use as guidance in evaluating recoveries. These limits are sufficient for evaluating the effect indicated for the individual sample results.

16.13.2 Corrective Action Poor surrogate recovery should be followed by re-analyzing a smaller aliquot to mitigate any matrix interferences. Evaluate the out of control surrogate for the effect on individual sample results.

16.14 Method Reporting Limit Check Standard

16.14.1 Acceptance Criteria Per client requirements or if the CCV is biased low for any compound, then evaluate the MRL check standard. Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio). Also, a percent difference +/-50% is recommended.

16.15 Sample Holding Time Expired

The customer is to be notified that the sample's holding time was missed and the customer is to decide if the sample analysis is to continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual for the appropriate data qualifier.

18.2 Initial Calibration and/or Initial Calibration Verification

All results reported with an unacceptable ICAL must be reported as estimated and all data shall be reported using defined qualifiers or flags or explained in the case narrative accordingly.

18.3 Continuing Calibration Verification

All results associated with an unacceptable CCV (other than #1 below) must be reported with the appropriate data qualifier, flag and/or explained in the case narrative.

1. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
2. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples with detects, then those detects must be reported with a qualifier, flag and/or explained in the case narrative.
3. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects must be reported with qualifiers, flags and/or explained in the case narrative as having less certainty. However, along with the data qualifiers, the case narrative may include information stating the fact that the results were not significantly affected if:
   a. *An MRL check standard was analyzed and found to be acceptable. The MRL must be the same as that analyzed in the MRL check standard for those analytes that were biased low in the CCV. Adjust MRLs (if required), flag data and state the certainty in the case narrative where the sensitivity of the instrument was demonstrated at the MRL; therefore, results were not significantly affected.*
   b. *With the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative and note procedure in case narrative.*
4. If the acceptance criteria was exceeded (biased high) for the CCV and there were detectable results in a sample, the results may be "qualified" if the results exceeded the regulatory/decision limit (this is to be stated in the case narrative along with the data qualifiers or flags).

18.4 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

18.5 Laboratory Control Sample

All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

18.6 Surrogate

Report sample results with the appropriate data qualifier.

18.7 Laboratory Duplicate

All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

18.8 Internal Standard

All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

18.9 Estimated Sample Results

18.9.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this SOP.

18.9.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

18.9.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty by reporting with the appropriate data qualifier.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

19.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 and 2A for both SCAN and SIM modes and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

19.3 Accuracy and Precision

Refer to Section 11.4 in the referenced method for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the electronic TO-15 Method Manual. Refer to Section 11.1.4.2 for the accuracy and precision requirements for concentrations at the LOQ/MRL.

19.4 Selectivity

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample

injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 98 or newer version
- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

19.5 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Therefore, ongoing demonstration of capable shall be performed and documented; however, the initial demonstration of method capability is not required.

19.6 Proficiency Testing (PT) Program

The laboratory shall participate in an air and emissions PT study for TO-15. The testing shall be performed in accordance with this document and meet the frequency and proficiency requirements detailed in the DoD QSM Version 5.0.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
22.0	03/21/15	C. Humphrey	Section 1 – Removed Note 1
			Section 4 – Revised section to include Hazard Assessment table
			Section 12.9 – Added Note 2
			Table 2A – Updated
			Table 3 – Updated
			Table 3A – Updated
			Table 4 – Updated
			Table 4A – Updated
			Attachment 3 – Added MAPH to Daily QC and Sample Review Checklists

21) References and Related Documents

21.1 EPA Method TO-14A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.2 EPA Method TO-15, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.3 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, January 1999.

21.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Addendum, January 17, 2002.

21.5 National Environmental Laboratory Accreditation Conference, *2003 NELAC Standard*, June 5, 2003, EPA 600/R-04/003 and 2009 TNI Standards.

21.6 *Preparation of Gas Phase Standards for Ambient Air Analysis,* Tekmar-DOHRMANN Application Note, Spring 96, Vol. 6.5.

21.7 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.

21.8 Arizona Administrative Code, Title 9. Health Services, Chapter 14. Department of Health Services Laboratories, December 31, 2006.

21.9 Florida Department of Environmental Protection, Chapter 62-160.

21.10 Minnesota Department of Health, 4740.2065, *Standard Operating Procedures*, Statutory Authority: MS s 144.97; 144.98; History: 31 SR 446, Posted: October 09, 2006, Revised April 16, 2010.

22) Appendix

22.1 Tables

Table 1: Instrument Tune Check Ion Abundance Criteria (TO-15)

Table 1A: Instrument Tune Check Ion Abundance Criteria (TO-14A)

Table 2: Volatile Organic Compounds, EPA Compendium Method TO-15 (SCAN)

Table 2A: Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)

Table 3: Standard Concentrations (SCAN) (Primary Sources)

Table 3A: Standard Concentrations (SIM) (Primary Sources)

Table 4: Standard Concentrations (SCAN) (Secondary Sources)

Table 4A: Standard Concentrations (SIM) (Secondary Sources)

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Daily QC and Sample Review Checklists

Attachment 4 – State and Project Specific Requirements

TABLE 1

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-15

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1] All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 1A

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-14A

Mass	Ion Abundance Criteria
50	15 to 40 percent of m/e 95
75	30 to 60 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5 to 9 Percent of m/e 95
173	Less than 2 Percent of m/e 174
174	>50 Percent of m/e 95
175	5 to 9 Percent of m/e 174
176	>95 and <101 Percent of m/e 174
177	5 to 9 Percent of m/e 176

Note: The criteria listed in Tables 1 and 1A shall be met or exceeded in order for EPA Compendium Methods TO-15 or TO-14A to be referenced.

TABLE 2 - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Bromochloromethane (IS1)	74-97-5	-	-	130	128, 132	-	-	-
Propene	115-07-1	42.08	NA	42	39,41	0.50	0.14	IS1
Dichlorodifluoromethane (CFC 12)	75-71-8	120.9	1.329	85	87, 101, 103	0.50	0.17	IS1
Chloromethane	74-87-3	50.49	0.911	50	52	0.50	0.15	IS1
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	76-14-2	170.9	1.455	135	137	0.50	0.19	IS1
Vinyl Chloride	75-01-4	62.50	0.9106	62	64	0.50	0.17	IS1
1,3-Butadiene	106-99-0	54.09	0.6149	54	39, 53	0.50	0.22	IS1
Bromomethane	74-83-9	94.94	1.6755	94	96	0.50	0.19	IS1
Chloroethane	75-00-3	64.52	0.8902	64	66	0.50	0.17	IS1
Ethanol	64-17-5	46.07	0.7893	45	46	5.0	0.80	IS1
Acetonitrile	75-05-8	41.05	0.7857	41	40	0.50	0.18	IS1
Acrolein	107-02-8	56.06	0.840	56	55	2.0	0.17	IS1
Acetone	67-64-1	58.08	0.7845	58	43	5.0	0.77	IS1
Trichlorofluoromethane	75-69-4	137.4	NA	101	103	0.50	0.17	IS1
Isopropyl Alcohol	67-63-0	60.10	0.7809	45	43	5.0	0.42	IS1
Acrylonitrile	107-13-1	53.06	0.8060	53	52	0.50	0.17	IS1
1,1-Dichloroethene	75-35-4	96.94	1.213	96	61	0.50	0.17	IS1
tert-Butanol	75-65-0	74.12	0.7887	59	57,41,43	1.0	0.33	IS1
Methylene Chloride	75-09-2	84.94	1.3266	84	49	0.50	0.17	IS1
Allyl Chloride	107-05-1	76.53	0.9376	41	76	0.50	0.16	IS1
Trichlorotrifluoroethane	76-13-1	187.38	1.5635	151	101	0.50	0.17	IS1

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Carbon Disulfide	75-15-0	76.14	1.2632	76	78	5.0	0.15	IS1
trans-1,2-Dichloroethene	156-60-5	96.94	1.2565	61	96	0.50	0.19	IS1
1,1-Dichloroethane	75-34-3	98.96	1.1757	63	65	0.50	0.16	IS1
Methyl tert-Butyl Ether	1634-04-4	88.15	0.7402	73	57	0.50	0.17	IS1
Vinyl Acetate	108-05-4	86.09	0.9317	86	43	5.0	0.65	IS1
2-Butanone (MEK)	78-93-3	72.11	0.7999	72	43	5.0	0.21	IS1
cis-1,2-Dichloroethene	156-59-2	96.94	1.2837	61	96	0.50	0.16	IS1
Diisopropyl Ether	108-20-3	102.18	0.7241	87	45,59,43	0.50	0.19	IS1
Ethyl Acetate	141-78-6	88.106	0.9003	61	70	1.0	0.35	IS1
n-Hexane	110-54-3	86.18	0.6548	57	86	0.50	0.15	IS1
Chloroform	67-66-3	119.4	1.4832	83	85	0.50	0.17	IS1
1,2-Dichloroethane-d4(S)	17060-07-0	-	-	65	67	-	-	IS1
Tetrahydrofuran	109-99-9	72.11	0.8892	72	71,42	0.50	0.20	IS1
Ethyl tert-Butyl Ether	637-92-3	102.176	0.7519	87	59,57	0.50	0.18	IS1
1,2-Dichloroethane	107-06-2	98.96	1.2351	62	64	0.50	0.16	IS1
1,4-Difluorobenzene(IS2)	540-36-3	-	-	114	88	-	-	-
1,1,1-Trichloroethane	71-55-6	133.4	1.3390	97	99, 61	0.50	0.17	IS2
Isopropyl acetate	108-21-4	102.13	0.8718	61	87,43	1.0	0.32	IS2
1-Butanol	71-36-3	74.1224	0.8098	56	41	1.0	0.48	IS2
Benzene	71-43-2	78.11	0.8765	78	77	0.50	0.16	IS2
Carbon Tetrachloride	56-23-5	153.8	1.5940	117	119	0.50	0.15	IS2

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Cyclohexane	110-82-7	84.16	0.7739	84	69,56	1.0	0.29	IS2
tert-Amyl Methyl Ether	994-05-8	102.176	0.7703	73	87,55,43	0.50	0.15	IS2
1,2-Dichloropropane	78-87-5	113	1.1560	63	62	0.50	0.16	IS2
Bromodichloromethane	75-27-4	163.8	1.980	83	85	0.50	0.15	IS2
Trichloroethene	79-01-6	131.4	1.4642	130	132	0.50	0.14	IS2
1,4-Dioxane	123-91-1	88.11	1.0337	88	58	0.50	0.16	IS2
Isooctane	540-84-1	114.23	0.6877	57	41	0.50	0.15	IS2
Methyl Methacrylate	80-62-6	100.12	0.944	100	69	1.0	0.31	IS2
n-Heptane	142-82-5	100.2	0.6837	71	57,100	0.50	0.17	IS2
cis-1,3-Dichloropropene	10061-01-5	111	1.224	75	77	0.50	0.14	IS2
4-Methyl-2-Pentanone	108-10-1	100.2	0.7965	58	85	0.50	0.16	IS2
trans-1,3-Dichloropropene	10061-02-6	111	1.217	75	77	0.50	0.16	IS2
1,1,2-Trichloroethane	79-00-5	133.4	1.4397	97	83	0.50	0.16	IS2
Chlorobenzene-d5(IS3)	3114-55-4	-	-	82	117	-	-	-
Toluene-d8(S)	2037-26-5	-	-	98	100	-	-	IS3
Toluene	108-88-3	92.14	0.8669	91	92	0.50	0.17	IS3
2-Hexanone	591-78-6	100.16	0.8113	43	58	0.50	0.16	IS3
Dibromochloromethane	124-48-1	208.3	2.451	129	127	0.50	0.16	IS3
1,2-Dibromoethane	106-93-4	187.9	2.1791	107	109	0.50	0.16	IS3
n-Butyl Acetate	123-86-4	116.16	0.8825	43	56, 73	0.50	0.16	IS3
n-Octane	111-65-9	114.23	0.6986	57	114	0.50	0.18	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Tetrachloroethene	127-18-4	165.8	1.6227	166	164	0.50	0.14	IS3
Chlorobenzene	108-90-7	112.6	1.1058	112	114	0.50	0.16	IS3
Ethylbenzene	100-41-4	106.2	0.8670	91	106	0.50	0.16	IS3
m-, p-Xylenes	179601-23-1	106.2	0.8642, 0.8611	91	106	1.0	0.30	IS3
Bromoform	75-25-2	252.8	2.899	173	175	0.50	0.15	IS3
Styrene	100-42-5	104.1	0.9060	104	78, 103	0.50	0.15	IS3
o-Xylene	95-47-6	106.2	0.8802	91	106	0.50	0.15	IS3
n-Nonane	111-84-2	128.26	0.7176	43	57, 85	0.50	0.15	IS3
1,1,2,2-Tetrachloroethane	79-34-5	167.9	1.5953	83	85	0.50	0.15	IS3
4-Bromofluorobenzene(S)	460-00-4	-	-	174	176	-	-	IS3
Cumene	98-82-8	120.2	0.8618	105	120	0.50	0.15	IS3
alpha-Pinene	80-56-8	136.24	0.8582	93	77	0.50	0.14	IS3
n-Propylbenzene	103-65-1	120.1938	0.8670	91	120,65	0.50	0.16	IS3
3-Ethyltoluene	620-14-4	120.2	0.8645	105	120	0.50	0.15	IS3
4-Ethyltoluene	622-96-8	120.2	0.8614	105	120	0.50	0.16	IS3
1,3,5-Trimethylbenzene	108-67-8	120.2	0.8652	105	120	0.50	0.16	IS3
alpha-Methylstyrene	98-83-9	118.19	0.9106	118	103,117	0.50	0.15	IS3
2-Ethyltoluene	611-14-3	120.2	0.8807	105	120	0.50	0.15	IS3
1,2,4-Trimethylbenzene	95-63-6	120.2	0.8758	105	120	0.50	0.15	IS3
n-Decane	124-18-5	142.28	0.7300	57	71,85	0.50	0.16	IS3
Benzyl Chloride	100-44-7	126.59	1.1004	91	126	0.50	0.11	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m³)	MDL[3] (µg/m³)	IS[4]
1,3-Dichlorobenzene	541-73-1	147	1.2884	146	148	0.50	0.15	IS3
1,4-Dichlorobenzene	106-46-7	147	1.2475	146	148	0.50	0.14	IS3
sec-Butylbenzene	135-98-8	134.2206	0.8601	105	134,91	0.50	0.16	IS3
p-Isopropyltoluene	99-87-6	134.2206	0.8573	119	134,91	0.50	0.15	IS3
1,2,3-Trimethylbenzene	526-73-8	120.1938	0.8944	105	120	0.50	0.15	IS3
1,2-Dichlorobenzene	95-50-1	147	1.3059	146	148	0.50	0.15	IS3
d-Limonene	5989-27-5	136.24	0.8402	68	93	0.50	0.14	IS3
1,2,Dibromo-3-Chloropropane	96-12-8	236.33	2.093	157	75, 39	0.50	0.099	IS3
n-Undecane	1120-21-4	156.31	0.7402	57	71, 85	0.50	0.15	IS3
1,2,4-Trichlorobenzene	120-82-1	181.5	1.459	180	182, 184	0.50	0.16	IS3
Naphthalene	91-20-3	128.17	1.0253	128	129	0.50	0.18	IS3
n-Dodecane	112-40-3	170.34	0.7487	57	71,85	0.50	0.13	IS3
Hexachlorobutadiene	87-68-3	260.8	1.556	225	227	0.50	0.14	IS3
Cyclohexanone	108-94-1	98.14	0.9478	55	42, 98	0.50	0.12	IS3
tert-Butylbenzene	98-06-6	134.22	0.867	119	134	0.50	0.15	IS3
n-Butylbenzene	104-51-8	134.22	0.867	91	134	0.50	0.17	IS3

(S) = Surrogate (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
NA = Not Available

Note 1: Additional compounds may be reported as long as the minimum requirements of this document are met. The compounds listed in this table are reported using TO-15 SCAN. The Selected Ion Monitoring (SIM) compounds are a subset of this list and are included in Table 2A.

Note 2: These are suggested primary and secondary ions. However, any ions in the analyte spectra that are sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 3: The laboratory performs three concentration level analyses (SIM, SCAN and Low Level SCAN). The method reporting limit listed is the standard SCAN limit (at or above lowest concentration in the initial calibration curve), but may change with each new initial calibration performed. Therefore, current reporting limits for the three analysis levels, MRLs in ppbv, and those from the Low Level SCAN should be reviewed in the electronic TO-15 Method Manual.

Note 4: The listing of the internal standard by which the compounds are quantitated is for TO-15 SCAN only. SIM compounds (SCAN subset) and their corresponding ions and internal standards are listed in Table 2A.

Note 5: m/e 101 is ~10% or less of m/e 85 (the base peak) and may not be present for low level results. Retention times must be carefully verified.

Table 2A – Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)					
Compound	**Primary Ion[1]**	**Secondary Ion[1]**	**MRL[2] (ug/m3)**	**MDL[2] (ug/m3)**	**IS**
Dichlorodifluoromethane	85	87	0.025	0.017	IS1
Chloromethane	52	50	0.025	0.019	IS1
Vinyl Chloride	62	64	0.025	0.0076	IS1
1,3-Butadiene	54	39	0.025	0.014	IS1
Bromomethane	94	96	0.025	0.0093	IS1
Chloroethane	64	66	0.025	0.0085	IS1
Acrolein	56	55	0.20	0.039	IS1
Acetone	58	43	2.5	0.056	IS1
Freon 11	101	103	0.025	0.015	IS1
1,1-Dichloroethene	96	98,61	0.025	0.0086	IS1
Methylene Chloride	84	49	0.10	0.013	IS1
Trichlorotrifluoroethane	151	153	0.025	0.0089	IS1
trans-1,2-Dichloroethene	96	98,61	0.025	0.0073	IS1
1,1-Dichloroethane	63	65	0.025	0.0061	IS1
Methyl tert-Butyl Ether	73	57	0.025	0.0093	IS1
cis-1,2-Dichloroethene	96	98,61	0.025	0.0092	IS1
Chloroform	83	85	0.10	0.018	IS1
1,2-Dichloroethane	62	64	0.025	0.0084	IS1
1,1,1-Trichloroethane	97	99	0.025	0.0059	IS1
Benzene	78	77	0.075	0.020	IS1
Carbon Tetrachloride	117	119	0.025	0.012	IS1
1,2-Dichloropropane	63	62,76	0.025	0.0073	IS2
Bromodichloromethane	83	85	0.025	0.0069	IS2
Trichloroethene	130	132	0.025	0.0085	IS2
1,4-Dioxane	88	58	0.10	0.0085	IS2
cis-1,3-Dichloropropene	75	77,39	0.025	0.0062	IS2
trans-1,3-Dichloropropene	75	77,39	0.025	0.0055	IS2
1,1,2-Trichloroethane	83	97,61	0.10	0.0079	IS2
Toluene	91	92	0.10	0.011	IS2
Dibromochloromethane	129	127	0.025	0.0088	IS3
1,2-Dibromoethane	107	109	0.025	0.0079	IS2
Tetrachloroethene	166	164	0.025	0.0082	IS2
Chlorobenzene	112	114	0.10	0.0092	IS3
Ethylbenzene	91	106	0.10	0.0097	IS3
m-&-p-Xylene	91	106	0.10	0.019	IS3
Styrene	104	103	0.10	0.0074	IS3
o-Xylene	91	106	0.10	0.0089	IS3
1,1,2,2-Tetrachloroethane	83	85	0.025	0.0072	IS3
1,3,5-Trimethylbenzene	105	120	0.10	0.0073	IS3
1,2,4-Trimethylbenzene	105	120	0.10	0.0083	IS3
1,3-Dichlorobenzene	146	148	0.025	0.0085	IS3
1,4-Dichlorobenzene	146	148	0.025	0.0081	IS3
1,2-Dichlorobenzene	146	148	0.025	0.0083	IS3
1,2-Dibromo-3-chloropropane	157	75	0.10	0.0095	IS3
1,2,4-Trichlorobenzene	182	184	0.025	0.013	IS3
Naphthalene	128	129	0.10	0.016	IS3
Hexachlorobutadiene	225	227	0.025	0.0092	IS3

NA = Not Available
(IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
Note 1: These are suggested primary and secondary ions. However, any ions in the analyte spectra that is sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 2: The method reporting limit listed is the standard SIM limit (lowest concentration in the initial calibration curve; must be higher than MDL), but may change with each new initial calibration performed. Therefore, current reporting limits should be reviewed. MDLs in ppbV may be reviewed in the electronic TO-15 Method Manual.

Table 3
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Bromochloromethane (IS1)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Propene	0.0792	0.198	0.396	0.99	4.95	24.75	49.5	99
Dichlorodifluoromethane (CFC 12)	0.0760	0.190	0.380	0.95	4.75	23.75	47.5	95
Chloromethane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
Vinyl Chloride	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
1,3-Butadiene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
Bromomethane	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
Chloroethane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
Ethanol	0.4128	1.032	2.064	5.16	25.80	129.00	258.0	516
Acetonitrile	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
Acrolein	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Acetone	0.4368	1.092	2.184	5.46	27.30	136.50	273.0	546
Trichlorofluoromethane	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Isopropyl Alcohol	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
Acrylonitrile	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,1-Dichloroethene	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
tert-Butanol	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
Methylene Chloride	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Allyl Chloride	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Trichlorotrifluoroethane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Carbon Disulfide	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
trans-1,2-Dichloroethene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,1-Dichloroethane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Methyl tert-Butyl Ether	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Vinyl Acetate	0.4200	1.050	2.100	5.25	26.25	131.25	262.5	525
2-Butanone (MEK)	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
cis-1,2-Dichloroethene	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
Diisopropyl Ether	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
Ethyl Acetate	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
n-Hexane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Chloroform	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
1,2-Dichloroethane-d4 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Tetrahydrofuran	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
Ethyl tert-Butyl Ether	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,2-Dichloroethane	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,4-Difluorobenzene(IS2)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
1,1,1-Trichloroethane	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
Isopropyl acetate	0.1832	0.458	0.916	2.29	11.45	57.25	114.5	229
1-Butanol	0.1824	0.456	0.912	2.28	11.40	57.00	114.0	228

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Benzene	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Carbon Tetrachloride	0.0920	0.230	0.460	1.15	5.75	28.75	57.5	115
Cyclohexane	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
tert-Amyl Methyl Ether	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
1,2-Dichloropropane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Bromodichloromethane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Trichloroethene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,4-Dioxane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Isooctane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Methyl Methacrylate	0.1712	0.428	0.856	2.14	10.70	53.50	107.0	214
n-Heptane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
cis-1,3-Dichloropropene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
4-Methyl-2-Pentanone	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
trans-1,3-Dichloropropene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,1,2-Trichloroethane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Chlorobenzene-d5 (IS3)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene-d8 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
2-Hexanone	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
Dibromochloromethane	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
1,2-Dibromoethane	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
n-Butyl Acetate	0.0928	0.232	0.464	1.16	5.80	29.00	58.0	116
n-Octane	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
Tetrachloroethene	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
Chlorobenzene	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
Ethylbenzene	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
m- & p-Xylene	0.1728	0.432	0.864	2.16	10.80	54.00	108.0	216
Bromoform	0.0912	0.228	0.456	1.14	5.70	28.50	57.0	114
Styrene	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
o-Xylene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
n-Nonane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,1,2,2-Tetrachloroethane	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
4-Bromofluorobenzene (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Cumene	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
alpha-Pinene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
n-Propylbenzene	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
3-Ethyltoluene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
4-Ethyltoluene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,3,5-Trimethylbenzene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
alpha-Methylstyrene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
2-Ethyltoluene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,2,4-Trimethylbenzene	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
n-Decane	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
Benzyl Chloride	0.0912	0.228	0.456	1.14	5.70	28.50	57.0	114
1,3-Dichlorobenzene	0.0912	0.228	0.456	1.14	5.70	28.50	57.0	114
1,4-Dichlorobenzene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
sec-Butylbenzene	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
p-Isopropyltoluene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
1,2,3-Trimethylbenzene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,2-Dichlorobenzene	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
d-Limonene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
1,2-Dibromo-3-Chloropropane	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
n-Undecane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,2,4-Trichlorobenzene	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Naphthalene	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
n-Dodecane	0.0904	0.226	0.452	1.13	5.65	28.25	56.5	113
Hexachlorobutadiene	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
Methacrylonitrile	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Cyclohexanone	0.0944	0.236	0.472	1.18	5.90	29.50	59.0	118
tert-Butylbenzene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
n-Butylbenzene	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 3A - Standard Concentrations (SIM) (Primary Sources)[1]

Compound Name	10pg	20pg	50pg	100pg	500pg	1000pg	2500pg	10,000pg	20,000pg	50,000pg
Freon-12	9.50	19.00	47.50	95.0	475	950	2375	9500	19000	47500
Chloromethane	10.10	20.20	50.50	101.0	505	1010	2525	10100	20200	50500
Vinyl Chloride	10.00	20.00	50.00	100.0	500	1000	2500	10000	20000	50000
1,3-Butadiene	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
Bromomethane	10.20	20.40	51.00	102.0	510	1020	2550	10200	20400	51000
Chloroethane	10.10	20.20	50.50	101.0	505	1010	2525	10100	20200	50500
Acrolein	11.30	22.60	56.50	113.0	565	1130	2825	11300	22600	56500
Acetone	54.60	109.20	273.00	546.0	2730	5460	13650	54600	109200	273000
Freon-11	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
1,1-Dichloroethene	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
Methylene Chloride	11.30	22.60	56.50	113.0	565	1130	2825	11300	22600	56500
Freon-113	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
trans-1,2-Dichloroethene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,1-Dichloroethane	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
Methyl tert-Butyl Ether	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
cis-1,2-Dichloroethene	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
Chloroform	11.20	22.40	56.00	112.0	560	1120	2800	11200	22400	56000
1,2-Dichloroethane	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
1,1,1-Trichloroethane	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
Benzene	11.30	22.60	56.50	113.0	565	1130	2825	11300	22600	56500
Carbon Tetrachloride	11.50	23.00	57.50	115.0	575	1150	2875	11500	23000	57500
1,2-Dichloropropane	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
Bromodichloromethane	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
Trichloroethene	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
1,4-Dioxane	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
cis-1,3-Dichloropropene	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
trans-1,3-Dichloropropene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,1,2-Trichloroethane	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
Toluene	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
Dibromochloromethane	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
1,2-Dibromoethane	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
Tetrachloroethene	10.10	20.20	50.50	101.0	505	1010	2525	10100	20200	50500
Chlorobenzene	11.10	22.20	55.50	111.0	555	1110	2775	11100	22200	55500
Ethylbenzene	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
m,p-Xylenes	21.60	43.20	108.00	216.0	1080	2160	5400	21600	43200	108000
Styrene	11.20	22.40	56.00	112.0	560	1120	2800	11200	22400	56000
o-Xylene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,1,2,2-Tetrachloroethane	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
1,3,5-Trimethylbenzene	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
1,2,4-Trimethylbenzene	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
1,3-Dichlorobenzene	11.40	22.80	57.00	114.0	570	1140	2850	11400	22800	57000
1,4-Dichlorobenzene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,2-Dichlorobenzene	11.10	22.20	55.50	111.0	555	1110	2775	11100	22200	55500
1,2-Dibromo-3-chloropropane	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
1,2,4-Trichlorobenzene	11.30	22.60	56.50	113.0	565	1130	2825	11300	22600	56500
Naphthalene	11.10	22.20	55.50	111.0	555	1110	2775	11100	22200	55500
Hexachloro-1,3-butadiene	11.20	22.40	56.00	112.0	560	1120	2800	11200	22400	56000

Note 1: The concentrations detailed in Table 3A may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4 - Standard Concentrations (SCAN) (Secondary Sources)[1]

Compound Name	25ng	Compound Name	25ng	Compound Name	25ng
Bromochloromethane (IS1)	**25.0**	1,1,1-Trichloroethane	26.00	alpha-Pinene	26.00
Propene	25.00	Isopropyl acetate	54.50	n-Propylbenzene	25.25
Dichlorodifluoromethane (CFC 12)	25.50	1-Butanol	55.75	3-Ethyltoluene	26.50
Chloromethane	24.75	Benzene	27.50	4-Ethyltoluene	26.50
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	25.75	Carbon Tetrachloride	26.75	1,3,5-Trimethylbenzene	26.50
Vinyl Chloride	25.25	Cyclohexane	52.75	alpha-Methylstyrene	26.00
1,3-Butadiene	26.75	tert-Amyl Methyl Ether	26.25	2-Ethyltoluene	26.25
Bromomethane	25.25	1,2-Dichloropropane	26.50	1,2,4-Trimethylbenzene	26.25
Chloroethane	25.25	Bromodichloromethane	27.00	n-Decane	25.75
Ethanol	127.25	Trichloroethene	26.00	Benzyl Chloride	27.25
Acetonitrile	25.50	1,4-Dioxane	27.25	1,3-Dichlorobenzene	27.25
Acrolein	26.75	Isooctane	26.00	1,4-Dichlorobenzene	26.50
Acetone	135.00	Methyl Methacrylate	52.50	sec-Butylbenzene	26.75
Trichlorofluoromethane	24.75	n-Heptane	26.75	p-Isopropyltoluene	25.25
Isopropyl Alcohol	52.50	cis-1,3-Dichloropropene	28.25	1,2,3-Trimethylbenzene	26.25
Acrylonitrile	26.00	4-Methyl-2-Pentanone	27.25	1,2-Dichlorobenzene	26.75
1,1-Dichloroethene	26.75	trans-1,3-Dichloropropene	27.00	d-Limonene	26.25
tert-Butanol	52.75	1,1,2-Trichloroethane	26.50	1,2-Dibromo-3-Chloropropane	25.75
Methylene Chloride	27.00	**Chlorobenzene-d5 (IS3)**	**25.0**	n-Undecane	25.25
Allyl Chloride	27.25	**Toluene-d8 (S)**	**25.0**	1,2,4-Trichlorobenzene	26.25
Trichlorotrifluoroethane	27.00	Toluene	26.50	Naphthalene	24.50
Carbon Disulfide	24.50	2-Hexanone	27.75	n-Dodecane	25.25
trans-1,2-Dichloroethene	26.50	Dibromochloromethane	27.50	Hexachlorobutadiene	26.75
1,1-Dichloroethane	26.00	1,2-Dibromoethane	27.00	Methacrylonitrile	26.00
Methyl tert-Butyl Ether	26.50	Butyl Acetate	28.00	Cyclohexanone	27.75
Vinyl Acetate	128.00	n-Octane	26.00	tert-Butylbenzene	26.50
2-Butanone (MEK)	27.00	Tetrachloroethene	24.75	n-Butylbenzene	27.25
cis-1,2-Dichloroethene	26.75	Chlorobenzene	27.00		
Diisopropyl Ether	27.25	Ethylbenzene	26.50		
Ethyl Acetate	53.50	m- & p-Xylene	52.50		
n-Hexane	26.25	Bromoform	27.00		
Chloroform	27.00	Styrene	27.25		
1,2-Dichloroethane-d4 (S)	**25.0**	o-Xylene	25.75		
Tetrahydrofuran	25.75	n-Nonane	25.50		
Ethyl tert-Butyl Ether	26.50	1,1,2,2-Tetrachloroethane	25.25		
1,2-Dichloroethane	26.25	**4-Bromofluorobenzene (S)**	**25.0**		
1,4-Difluorobenzene(IS2)	**25.0**	Cumene	25.50		

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4A – ICV/LCS Standard Concentrations (SIM) (Secondary Sources)[1]

Compound Name	500pg
Freon-12	510
Chloromethane	495
Vinyl Chloride	505
1,3-Butadiene	535
Bromomethane	505
Chloroethane	505
Acrolein	535
Acetone	2700
Freon-11	495
1,1-Dichloroethene	535
Methylene Chloride	540
Freon-113	540
trans-1,2-Dichloroethene	530
1,1-Dichloroethane	520
Methyl tert-Butyl Ether	530
cis-1,2-Dichloroethene	535
Chloroform	540
1,2-Dichloroethane	525
1,1,1-Trichloroethane	520
Benzene	550
Carbon Tetrachloride	535
1,2-Dichloropropane	530
Bromodichloromethane	540
Trichloroethene	520
1,4-Dioxane*	545
cis-1,3-Dichloropropene	565
trans-1,3-Dichloropropene	540
1,1,2-Trichloroethane	530
Toluene	530
Dibromochloromethane	550
1,2-Dibromoethane	540
Tetrachloroethene	495
Chlorobenzene	540
Ethylbenzene	530
m,p-Xylenes	1050
Styrene	545
o-Xylene	515
1,1,2,2-Tetrachloroethane	505
1,3,5-Trimethylbenzene	530
1,2,4-Trimethylbenzene	525
1,3-Dichlorobenzene	545
1,4-Dichlorobenzene	530
1,2-Dichlorobenzene	535
1,2-Dibromo-3-chloropropane	515
1,2,4-Trichlorobenzene	525
Naphthalene	490
Hexachloro-1,3-butadiene	535

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Attachment 1

Training Plan

Training Plan for Analysis of VOCs by GC/MS

Trainee _____ Trainer _____ Instrument _____ Training Completion Date _____

1. Read SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____

2. Read Methods TO-14A & TO-15A *Training Duration* _____ Trainer ____ Trainee ____ Date _____

3. Demonstrated understanding of the scientific basis of the analysis Trainer ____ Trainee ____ Date _____
   Whole air sample preconcentration techniques
   Gas chromatography *Training Duration* _____
   Mass spectrometry

4. Demonstrated familiarity with related SOPs Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences; Rev. ___
   SOP for Making Entries onto Analytical Records; Rev. ___ *Training Duration* _____
   SOP for Manual Integration Policy; Rev. ____
   SOP for Significant Figures; Rev. ____
   SOP for Nonconformance and Corrective Action; Rev. ____
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation; Rev. ____
   SOP for Cleaning and Certification of Summa Canisters; Rev. ____

5. Observe performance of SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant introduction (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

6. Perform SOP with supervision *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant use (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

7. Independent performance of the SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant proficiency (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)
   ___initial demonstration of competency (4 Laboratory Control Samples)

8. Instrument operation and maintenance Trainer ____ Trainee ____ Date _____
   ___autosampler *Training Duration* _____
   ___GC and capillary column installation *Training Duration* _____
   ___mass spectrometer *Training Duration* _____
   ___data system *Training Duration* _____

Attachment 2

Initial Calibration Checklist

Initial Calibration Review Checklist - EPA Compendium Method TO-15

ICAL Date:_____ ICAL ID:_____ LIMS ICAL ID:_____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS11 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan Scan Low Level (0.1ng): ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation in the ICAL file?	☐
	☐ BFB Tune analysis Report	☐
	☐ Calibration Status Report (aka Calibration History)	☐
	☐ Response Factor Report/Percent RSD	☐
	☐ Quantitation Report for each calibration standard (including manual integration documentation)	☐
	☐ ICV Quantitation Report	☐
	☐ TO-15 Standard Calculation Spreadsheet	☐
☐	2. Was the ICAL performed continuously (not interrupted for maintenance or sample analysis)?	☐
☐	3. Have all the calibration standards been analyzed within 24 hours of each other?	☐
☐	4. Does the BFB tune check standard analysis at the start meet the tune criteria?	☐
☐	5. Are all the analytes in the blank analysis <MRL?	☐
☐	6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels?	☐
☐	7. Were the standards analyzed from low concentration to high concentration?	☐
☐	8. For each analyte, are there no levels skipped?	☐
☐	9. For each analyte, is there only one value used for each calibration level?	☐
☐	10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL?	☐
☐	11. For each analyte, is the corresponding signal to noise ratio at least 3:1 at the lowest point on the curve?	☐
☐	12. For each analyte, are the corresponding upper levels free from saturation?	☐
☐	13. If a calibration level is dropped, are all the responses for each target analyte dropped and is the information noted in the ICAL explaining the reason?	☐
☐	14. Is the average RSD ≤30% for all analytes, with no more than two exceptions ≤40%?	☐
☐	15. Is the response Y at each calibration level within 40% of the mean area response over the initial calibration range for each internal standard?	☐
☐	16. Percent recovery for each analyte in the ICV 70%-130% (50-150% for VA, unless AFCEE or DoD)?	☐
☐	17. Was the RRT for each target compound at each calibration level within 0.06RRT units of the mean RRT for the compound?	☐
☐	18. Is the retention time shift for each of the internal standards at each calibration level within 20s of the mean retention time over the initial calibration range for each standard?	☐
☐	19. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages.	☐
☐	20. Is the ICAL good at 0.5ng (or 0.1ng)–100ng (Scan) or 10-20000pg (SIM) for all compounds? ☐ Yes ☐ No If not, note exceptions and the corresponding MRLs below – *Specify applicable range...*	☐
☐	21. Are ALL of the peak selections for each analyte correct according to retention time (all RTs must be checked by both the initial and peer reviewer)?	☐

COMMENTS:

Analyst:_____ Date: _____

Secondary Reviewer: _____ Date: _____

Attachment 3

Daily QC and Sample Review Checklists

Daily QC Review Checklist

(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

EPA Compendium Method TO-15

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation present? ☐ CORRECT BFB Tune analysis Report ☐ CCV analysis Quantitation Report & %D Report ☐ LCS analysis Quantitation Report ☐ MB analysis Quantitation Report	☐
☐	2. BFB **tune** check standard analysis meet the tune criteria for the method indicated above?	☐
☐	3. Analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**?	☐
☐	4. Does the **CCV** have a difference ≤30% for all analytes? [Note <u>all</u> outliers biased high and/or low]	☐
☐	5. All **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)?	☐
☐	6. All **IS** responses within ±40% of CCV or the midpoint in the ICAL?	☐
☐	7. All **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%)	☐
☐	8. All analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)?	☐
☐	9. **LCS** %R within the lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)?	☐
☐	10. All analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits?	☐

COMMENTS:

Air-Phase Petroleum Hydrocarbons

☐	1. Does the **CCV** meet the following criteria? • Percent difference ≤30%. • One compound or range can be >30%, but less than 50%. • No single analyte or range may be >50%. [Note outliers biased high and/or low]	☐
☐	2. Does lab **duplicate** meet an RPD of ≤30% for results >5x MRL? Repeat analysis if:	☐
☐	3. Are the analytes in the **LCS** within 70%-130% recovery?	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐ LIMS Run Approval ☐ LIMS Supervisor Approval

Analyst: _____ Secondary Reviewer: _____

Date: _____ Date: _____

Sample Review Checklist
(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

EPA Compendium Method TO-15

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____ **Project #:** _____

Instrument: ☐ MS3 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. All analyte hits in the samples within the **calibration range** and/or noted?	☐
☐	2. All **peak integrations** acceptable?	☐
☐	3. All **manual integrations** flagged and documented?	☐
☐	4. Have **Q values** been verified for each peak?	☐
☐	6. All **calculations** correct?	☐
☐	7. Has the analyst initialed and dated each **quantitation report**?	☐
☐	8. For **TICs** are the relative intensity and other requirements met?	☐
☐	9. **Auto report** correct?	☐
☐	10. **MRL** = _____ ☐ ng ☐ pg (ethanol, acetone, vinyl acetate = 5.0ng)	☐
☐	11. Pressurized with **Helium**? Is the worksheet completed for all samples?	☐
☐	12. Report to **MDL**? ☐ Yes ☐ No	☐
☐	13. **Global Minimum Detection Limit** = _____ ☐ ng ☐ pg	☐
☐	14. **DOD:** Are **manual integrations** notated in the **case narrative?**	☐

COMMENTS:

Air-Phase Petroleum Hydrocarbons

☐	1. Are all manual **integrations** flagged and documented (except for HC ranges)?	☐
☐	2. Are all peak **integrations** acceptable?	☐
☐	3. Has the analyst initialed and dated each **quantitation report**?	☐
☐	4. Are the associated ICAL responses correct?	☐
☐	5. Are the sample responses entered into the template correctly?	☐
☐	6. Are the TO-15 target compounds entered into the template correctly?	☐
☐	7. Does the lab **duplicate** meet a RPD of ≤30% for results >5x the MRL? Otherwise, repeat analyses if:	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐ LIMS Run Approval

Analyst: _____

Date: _____

☐ LIMS Supervisor Approval

Secondary Reviewer: _____

Date: _____

Attachment 4

State and Project Specific Requirements

Minnesota Requirements	
Item	**Criteria**
Holding Time (HT)	14 days
Tedlar bags	Not allowed for sampling or sample dilution
Canisters and flow controllers	Individually certified Individually leak checked before shipment
	Samples with concentrations outside of the calibration curve will have a zero canister analysis performed to check for carryover. If carryover is detected, system bake out shall be performed and documented. Additionally, in instances where the laboratory has evidence on file that a particular compound when present at a high concentration does not exhibit carry-over, the samples will not be reanalyzed. When samples are analyzed that have a higher concentration than the evidence on file, the above requirements must be followed. Also, samples that have hits below the MRL will not be reanalyzed when analyzed after a sample with concentrations over the calibration range.
Method Reporting Verification Check	Analyze a Method Reporting Verification at the beginning of the sequence prior to analyzing samples. Acceptance criteria ±40%.
Duplicates	10 percent laboratory duplicates
Record retention	MN/NELAC 5 years MPCA (Minnesota Pollution Control Agency) compliant samples 10 years
Tier level	TIII

Arizona Requirements	
Item	**Criteria**
LCS	70-130% (vinyl acetate 50-150%)

Department of Toxic Substances Control (DTSC) Requirements	
Item	**Criteria**
Holding Time (HT)	72 hour hold time for canisters

EPA Region 9 Requirements	
Item	**Criteria**
Holding Time (HT)	14 days

ATTACHMENT D

QUALITY CONTROL FORMS

FIELD ASSESSMENT CHECKLIST

		Yes	No	NA	Explain
A	**GENERAL PROCEDURES**				
A1	**PROJECT PLANS**				
	Is a copy of the approved Work Plan (WP) onsite and readily available to field personnel?				
	Is a copy of the approved Accident Prevention Plan (APP) onsite and readily available to field personnel?				
	Is a copy of the approved Quality Assurance Project Plan (QAPjP) onsite and readily available to field personnel?				
	Are field personnel knowledgeable of the project plans?				
A2	**ORGANIZATION, PERSONNEL, AND RESPONSIBILITIES**				
	Does either the WP or the QAPjP include an organizational chart?				
	Does either the WP or the QAPjP include a list of personnel responsibilities?				
	Is a daily meeting held to present the planned activities to the team and provide updates on any health and safety and Quality Control (QC)				
	Do the field personnel understand the chain of command? Discuss with individuals.				
	Have all field personnel reviewed and signed the APP?				
	Do field personnel have certification documentation on hand?				

		Yes	No	NA	Explain
	Is the site secure from unauthorized personnel?				
A3	**SAMPLE DOCUMENTATION AND HANDLING**				
	Is a Sample Manager/Custodian identified? Name:				
	Have all personnel involved with sample handling/shipment received training in all applicable and appropriate procedures?				
	Are all transfers of sample custody documented?				
	Have samples been preserved as specified in the approved QAPjP?				
	Does the identification and packaging of samples include each of the following items:				
	a. Entries in permanent ink of all applicable sample labels and tags, chain-of-custody (COC) forms and seals, and any associated paperwork?				
	b. Legible sample IDs				
	c. Complete and legible sample labels				
	d. Signed and dated COC forms				
	e. Placement of groundwater samples in a clear Ziploc plastic bag (if applicable)				
	Are sample collection logs and shipping records maintained onsite, well organized and accurate? Is any QC performed?				
	Are the coolers packed in a contamination-free area?				

		Yes	No	NA	Explain
A3	**SAMPLE DOCUMENTATION AND HANDLING (continued)**				
	Is there adequate protection against breakage of sample containers (for example packed with sufficient padding material)?				
	Are procedures in place to ensure that samples that need to be maintained at 4 degrees C are kept at that temperature and are not going to freeze or overheat?				
	Are all entries on the COC and associated paperwork (field forms, logbook, air bill) complete, accurate, legible and made in permanent ink where required?				
	Are samples shipped to the appropriate laboratories?				
	Are COC forms placed in a clear waterproof Ziploc plastic bag and taped to the inside of the cooler or box lid?				
	If coolers are shipped, are they properly secured with duct and clear/strapping tape?				
	Are shipping labels filled out properly?				
	Are shipping labels appropriately secured to the outside of the coolers? Additional clear tape is needed to secure self-adhesive air bill envelopes.				
	Are samples shipped to the laboratories in a timely fashion to minimize potential problems with holding times exceedances?				
	Is there a system in place to report sample shipments to the laboratory contact?				
	Is there a system in place to report sample shipments to project personnel not present onsite?				
	Are accurate sample collection logs and shipping records maintained onsite in a well-organized fashion?				

		Yes	No	NA	Explain
A4 FIELD RECORDS					
	Are daily field activity logs used and kept onsite?				
	Are all field activity logs dated and signed?				
	Are all entries in the log made promptly? Is time indicated in military format?				
	Are any blank pages or spaces left in the log? Any blank space in the log as well as the bottom of the last page should be crossed out, signed and dated.				
	Are all field log entries made in indelible ink?				
	Are all corrections indicated by a single-line strikethrough, dated and initialed?				
	Do all logs contain at least the following information on the cover:				
	a. Project name				
	b. Site name				
	c. Weather notes/visitors				
	d. Start and end dates for the field effort				
	e. Names of the individuals that are using the logbook?				
	Do all logs contain at least the following information on the cover:				
	Are entries in the logs adequate to allow a competent person other than the originator to reconstruct the activities?				
	Are sufficient data recorded to allow all field calculation to be replicated (for example total purge calculations for soil-vapor sampling)?				
	Are field calculations accurate? The inspector should verify 10% of the calculations.				
	Are soil-boring logs filled out promptly, accurately, and legibly?				

		Yes	No	NA	Explain
	Does sample collection information include the following items:				
	a. Sampling personnel				
	b. Sample identification				
	c. Sampling location map				
	d. Sample depth				
	e. Sample description				
	f. Collection date				
	g. Collection time (military time)				
	h. Ambient weather conditions				
	i. Analytical suite				
	j. Field data sheets				
	Are sample log sheets or electronic tracking file filled out promptly accurately and legibly?				
A5	**INSTRUMENT MAINTENANCE AND CALIBRATION**				
	Has all analytical and monitoring equipment been calibrated according to the schedule required in the approved WP or QAPjP?				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				

		Yes	No	NA	Explain
	Are all instruments In use (or that will be used today) within calibration tolerance? Inspector should request that the designated calibration personnel analyze the appropriate standard as an unknown.				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Instrument_____ Calibration Personnel _____				
	Are all instruments used properly, as detailed in the FSP or QAPjP and according to SOPs and manufacturer's instructions?				
	Are all instruments appropriately maintained, according to manufacturer's instructions?				
	Additional comments on section A.				
B	**FIELD PROCEDURES**				
B1	Has the inspector reviewed the WP and QAPjP prior to the site visit?				
B2	Do the WP or QAPjP include specific procedural instructions, necessary for the completion of the scheduled field activities?				
B3	Do the WP or QAPjP and SOPs provide sufficient detail and effectively describe the objectives and requirements of the activities?				
B4	Is all equipment necessary to complete the work at hand and in good working condition?				

		Yes	No	NA	Explain
B5	Are daily reports prepared to identify deviations from the approved plans or SOPs?				
B6	Have appropriate authorizations been granted on all the deviations?				
B7	Have all field and QC samples been collected as per the WP or QAPjP and applicable instructions? Inspector should check for proper sample collection order, for example volatile organic compounds (VOC) first, proper containers, no homogenization for samples to be analyzed for VOCs, etc.).				
B8	Were any samples collected in a fashion that may question their integrity; for example, VOCs collected in the vicinity of a running vehicle?				
B9	Additional comments on section B.				
C	**INSPECTION SUMMARY**				
C1	Do the responses to the inspector indicate that the field personnel are aware of the quality assure (QA)/QC and its importance to the success of the projects?				
C2	Do field personnel place positive emphasis on QA/QC procedures?				
C3	Have responses with respect to QA/QC procedures been open and direct?				
C4	Has a cooperative attitude been displayed by the field personnel?				
C5	Are all procedures and documentation performed consistent with the Work Plan or QAPjP? Is there evidence that the field team has corrected deficiencies identified in the previous inspection (if applicable)?				
C6	Additional comments on section C.				
D	**DEFICIENCY REPORT**				
D1	Were any deficiencies identified as a result of the field inspection?				
D2	Were all deficiencies and observations discussed with the field team leader during inspection debriefing?				

		Yes	No	NA	Explain
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				

		Yes	No	NA	Explain
D	**DEFICIENCY REPORT (continued)**				
D2	Were all deficiencies and observations discussed with the field team leader during inspection debriefing? (continued)				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				

		Yes	No	NA	Explain
D	**DEFICIENCY REPORT (Continued)**				
D2	Were all deficiencies and observations discussed with the field team leader during inspection debriefing? (Continued)				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				

		Yes	No	NA	Explain
D	**DEFICIENCY REPORT (Continued)**				
D2	Were all deficiencies and observations discussed with the field team leader during inspection debriefing? (Continued)				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				
	Deficiency # _____ Description _____ _____ _____ _____ Corrective Action(s): _____ _____ _____ _____ _____ Implementation Date(s):				

NON-CONFORMANCE REPORT

Project:		RFI No.:		Date:
To:		Contract No:		
		REFERENCES		
Attention:		Drawing/Spec:		
Subject:		Detail/Section:		
		Discipline:		

POTENTIAL IMPACT	ROUTING	DATE SENT	DATE REC'D	COMMENTS
☐ QUALITY/TECHNICAL COMPLETION				
☐ COST				
☐ SCHEDULE				
ACTIVITY:				
	RESPONSE REQUESTED BY:		PRIORITY:	

NONCONFORMANCE	
CORRECTIVE ACTION	

Addressee: Sign and return original to:	**By:**
	Name/ Signature:
	Title: