DEPARTMENT OF THE AIR FORCE
HEADQUARTERS 49TH WING (ACC)
HOLLOMAN AIR FORCE BASE, NEW MEXICO

25 February 2015

DeAnna Rothhaupt
Chief, Holloman AFB Environmental
550 Tabosa Avenue
Holloman AFB NM 88330-8458

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FEB 27 2015

NMED
Hazardous Waste Bureau

USEPA, Region 6 (6PD-F)
Attn: Mr. Chuck Hendrickson, Project Manager
1445 Ross Avenue, Suite 1200
Dallas, TX 75202-2733

Dear Mr. Hendrickson,

Holloman AFB is pleased to submit the Final Remedial Investigation Work Plan for SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range Munitions Response Sites, Holloman Air Force Base, NM.

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 fine and imprisonment for knowing violations.

If you have any questions regarding this submittal, please contact me at (575) 572-6675.

Sincerely,

DEANNA ROTHHAUPT, GS-12, DAFC

Attachment:
Final Remedial Investigation Work Plan for SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range Munition Response Sites, Holloman Air Force Base, New Mexico.

cc:

(w/Atch)	(w/Atch)	(w/o Atch)
Mr. David Strasser	Mr. John Kieling, Chief	Mr. Will Moats
Hazardous Waste Bureau	Hazardous Waste Bureau	Hazardous Waste Bureau
5500 San Antonio Dr, NE	2905 Rodeo Park Dr, East, Building 1	5500 San Antonio Dr, NE
Albuquerque NM 87109-4127	Santa Fe NM 87505-6303	Albuquerque NM 87109-4127

The information herein is For Official Use Only (FOUO) which must be protected under the Freedom of Information Act of 1966 and Privacy Act of 1974, as amended. Unauthorized disclosure or misuse of this PERSONAL INFORMATION may result in criminal and/or civil penalties.

FINAL

SR859a FORMER SKEET RANGE 2 and TS862a JEEP TARGET AREA SKEET RANGE

REMEDIAL INVESTIGATION WORK PLAN

UNIFORM FEDERAL POLICY QUALITY ASSURANCE PROJECT PLAN

HOLLOMAN AIR FORCE BASE
NEW MEXICO

Performance Based Remediation
Contract Number: FA8903-13-C-0008

Prepared for

AIR FORCE CIVIL ENGINEER CENTER
2261 Hughes Ave., Suite 155
Joint Base San Antonio Lackland, Texas 78236-9853

February 2015

Prepared by:

FPM Remediations, Inc.
584 Phoenix Drive
Rome, NY 13441

URS **Group, Inc.**
7720 N 16th Street, Suite 100
Phoenix, AZ 85020-4493

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UFP-QAPP for SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range

Remedial Investigation

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel 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 fine and imprisonment for knowing violations.

Sites:

SR859a Former Skeet Range 2 and
TS862a Jeep Target Area Skeet Range
Holloman Air Force Base
New Mexico

Author: Peter Corigliano III
Peter Corigliano

Title: Site Manager
Date:

Submitted to:
United States Environmental Protection Agency Region 6 (6PD-F)
Mr. Chuck Hendrickson
1445 Ross Ave, Suite 1200
Dallas TX 75202-2750

Reviewer: Maureen L. Whalen
Maureen Whalen PG, CPG, PMP

Title: Project Manager
Date:

Prepared for:

Air Force Civil Engineer Center (AFCEC)
2261 Hughes Ave., Suite 155
Joint Base San Anotonio Lackland,
Texas 78236-9853

Reviewer: Laura Peters
Laura Peters

Title: Environmental Restoration Manager
Date: 2/17/15

Prepared by:
PM Remediations, Inc.
584 Phoenix Drive, Rome, NY 13441

February 2015

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LIST OF ABBREVIATIONS AND ACRONYMS

µg/kg	microgram per kilogram
A	Analytical
AF	Air Force
AFCEC	Air Force Civil Engineer Center
bgs	below ground surface
BNA	Base, Neutral, and Acid
°C	degrees Celsius
CAS	Chemical Abstracts Service
CCV	Continuing Calibration Verification
CEIER	Civil Environmental and Infrastructure Engineering
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CES	Civil Engineer Squadron
CFR	Code of Federal Regulations
CO	Contracting Officer
CoC	Chain of Custody
COR	Contracting Officer Representative
CRP	Compliance Restoration Program
CSE	Comprehensive Site Evaluation
CZRX	Environmental Center of Excellence Restoration - Execution
DD	Decision Document
DERP	Defense Environmental Restoration Program
DFTPP	Decafluorotriphenylphosphine
DGPS	Differential Global Positioning System
DL	Detection Limit
DMM	Discarded Military Munitions
DoD	Department of Defense
DQCRs	Daily Quality Control Reports
DQI	Data Quality Indicator
DQO	Data Quality Objective
EE/CA	Engineering Evaluation/Cost Analysis
ERP	Environmental Restoration Program
ERPIMS	Environmental Restoration Program Information Management System
ESS	Enterprise Sourcing Squadron
FPM	FPM Remediations, Inc.
ft	foot or feet
g	gram(s)
H&S	Health and Safety
HAFB	Holloman Air Force Base
HSP	Health and Safety Plan
ICAL	Initial Calibration
ICP	Inductively Coupled Plasma
ICS	Interference Check Solutions
ICV	Initial Calibration Verification
ID	Identification

IDL	Instrument Detection Limit
IDW	Investigation Derived Waste
IRP	Installation Restoration Program
IS	Internal Standards
JBSA Lackland	Joint Base San Antonio Lackland
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
MC	Munitions Constituents
MDL	Method Detection Limit
MEC	Munitions and Explosives of Concern
mg/kg	milligram(s) per kilogram
mg/L	milligrams per liter
MMRP	Military Munitions Response Program
MRA	Munitions Response Area
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
N/A	Not Available
NCP	National Oil Hazardous Substances Pollution Contingency Plan
NFA	No Further Action
No.	Number
O&M	Operation and Maintenance
oz	ounce
PBR	Performance Based Remediation
PDS	Post Digestion Spike
PE	Performance Evaluation
PGM	Program Manager
pH	Measure of the acidity or basicity of a solution (pH = -log[hydrogen ion concentration])
PKB	Environmental Contracting
PM	Project Manager
PMP	Project Management Plan
POC	point of contact
PPE	Personal Protective Equipment
ppm	parts per million
PT	Proficiency Testing
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
r	Correlation Coefficient
RA	Removal Action
RBSL	Risk-Based Screening Level
RI	Remedial Investigation

RPD	Relative Percent Difference
RPM	Restoration Program Manager
RRT	Relative Retention Time
RSL	Regional Screening Level
S	Sampling
SARA	Superfund Amendments and Reauthorization Act
SC	Site Closeout
SI	Site Investigation
SIM	Selective Ion Monitoring
SoB	Statement of Basis
SOP	Standard Operating Procedure
SPLP	Synthetic Precipitation Leaching Procedure
SRM	Source Reference Materials
TBD	To Be Determined
TCLP	Toxicity Characteristic Leaching Procedure
TO	Task Order
U.S.	United States
USAF	United States Air Force
USEPA	United States Environmental Protection Agency
UFP-QAPP	Uniform Federal Policy – Quality Assurance Project Plan
UXO	Unexploded Ordnance
WP	Work Plan
XRF	X-ray fluorescence

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Executive Summary

Introduction

This Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) has been prepared in support of a Remedial Investigation (RI), which would provide sufficient information for remedial action, if necessary, to achieve site closeout (SC) at Holloman Air Force Base (HAFB) sites SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range (**Figure 1**). FPM Remediations, Inc. (FPM) has been contracted by the Air Force Civil Engineer Center (AFCEC) under Contract FA8903-13-C-0008 to conduct Performance Based Remediation (PBR), in compliance with U.S. Environmental Protection Agency (USEPA) and New Mexico Environment Department (NMED) regulatory requirements, at certain sites at HAFB.

The Air Force defines a site as having achieved SC when all active management and monitoring at the environmental cleanup site have been completed, no additional environmental cleanup funds will be expended at the site, and the Air Force has obtained regulatory concurrence. SC occurs when cleanup goals have been achieved in accordance with the final Decision Document (DD) (if regulatory structure(s) require a DD for the site) that allow unrestricted use of the property. That is, no further long term monitoring (LTM) and/or including institutional controls (ICs) are required by both the regulator and Air Force and that all site decommissioning has been completed. Achieving SC may include, but not be limited to, the dismantling, removal, recycling, reclamation and/or disposal of all remedial activity systems and ancillary equipment above and underground to return the site to its natural state, well abandonment, and site restoration in accordance with Base and regulatory requirements. Regulatory concurrence may include variations such as “no further active remediation”, “regulatory closure with restrictions”, “conditional site closure” or other functional equivalents. These variations would involve requirements for LTM and would therefore not allow for unrestricted use, and as such is not SC.

The RI for the SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range Sites are being performed under the United States Air Force (USAF) Military Munitions Response Program (MMRP) under the authority of the Defense Environmental Restoration Program (DERP) and in accordance with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980, as amended by the Superfund Amendments and Reauthorization Act (SARA) of 1986. The Department of Defense (DoD) has established the MMRP under the DERP to address DoD sites with unexploded ordnance (UXO), discarded military munitions (DMM), and munitions constituents (MC) located on current and former military installations.

Site Background

The SR859a Former Skeet Range 2 is located in the southeastern portion of the installation approximately 1,300 feet (ft) north of Forty Niner Avenue and 650 ft north of Holloman runway control tower. A cultural resource survey documented shotgun shell cases, burned clay pigeons, .45-caliber automatic pistol cartridges, and hundreds of spent cartridges located in three trash piles (USACE, 2013). Additionally, 1940’s historical aerial photos show a four firing point skeet range at this location. Dark areas in contrast to the natural terrain, indicative of clay pigeon

debris, are also visible on aerial photography from 1945. A Comprehensive Site Evaluation (CSE) Phase I investigation and CSE Phase II investigation were conducted at the SR859a Former Skeet Range site (Shaw, 2010). During visual reconnaissance surveys lead shot and clay target debris was observed during both investigations. During the CSE Phase II the site was surveyed utilizing X-ray fluorescence (XRF) screening of soil samples at 143 locations for lead and the collection of 24 soil samples for analysis of polynuclear aromatic hydrocarbon (PAHs) by a fixed base laboratory. The CSE Phase II XRF field activities indicated one XRF sample exceeded the USEPA Residential Regional Screening Level (RSL) for lead in soil (400 milligrams/kilograms [mg/kg]) and PAHs concentration in soil samples exceeded USEPA RSLs in 14 of 24 samples (20 surface soil samples were collected and two subsurface samples from 6-12 inches and two from 12 – 18 inches were collected). One sample from the 6-12 inch interval indicated exceedances and both samples from the 12-18 inch interval indicated exceedances. The results of the Human Health Risk Screening indicated potential risk to future residential receptors due to PAH concentrations in soil. The CSE Phase II report recommended splitting the Munitions Response Area (MRA) into two Munitions Response Sites (MRSs): SR859 (34.3 acres) was recommended for no further action (NFA) due to lack of Munitions and Explosives of Concern (MEC) and MC exceeding USEPA screening levels and SR859a (8.0 acres) was recommended for future munitions response actions. This UFP-QAPP will address only the 8 acres that was recommended for future response actions (designated as SR859a by the CSE Phase II Report) (**Figure 2**).

The land surface within the MRS is relatively flat open space consisting of desert scrubland. The Holding Area Munitions Storage occupies a small portion of land adjacent to the MRS to the north.

The TS862a Jeep Target Area Skeet Range is located just northeast of the active Jeep Target Area in the south central portion of the installation. The range was described by a Laboratory of Anthropology Site Record as a skeet or trap range in addition to the Jeep Target Area with firing positions (5ft by 6 ft concrete pads) and scattered clay target debris. The CSE Phase I visual reconnaissance survey found clay pigeon debris; lead shot; an area of burned shotgun casings; and small arms projectiles (USACE, 2013). Remains of a rock pathway likely connecting former firing positions for the skeet range were also discovered. A CSE Phase II investigation was also conducted at the site (HDR, 2013). During the investigation dense concentrations of clay target debris was identified along with shotgun shell waddings and lead shot. Surface soil samples for XRF screening were collected at 137 locations. The CSE Phase II field activities did not indicate that any of the XRF screening samples exceeded the USEPA Residential RSL for lead of 400 mg/kg. In addition a total of 41 locations (31 surface and 10 subsurface [5 from 6-12 inches below ground surface (bgs) and 5 from 12 – 18 inches bgs]) were sampled for PAHs. At least one PAH analyte exceeded its human health screening level in 16 of the samples taken. Two subsurface soil samples indicated exceedances from the 12 – 18 inches interval. Human Health Risk Screening indicated potential risks to future residential receptors from PAH contaminated soils (USACE, 2013). The CSE Phase II report recommended splitting the initial MRA into two MRSs: TS862 (34.6 acres) was recommended for NFA due to lack of MEC and MC exceeding USEPA residential RSLs and TS862a (5.7 acres) was recommended for future munitions response actions. This UFP-QAPP will address only the 5.7 acres that was recommended for future response actions (TS862a) (**Figure 3**).

The MRS is currently unused and the land surface is relatively flat open space with vegetation consistent with desert scrubland.

It should be noted that site-wide lead background concentrations information was obtained from the Basewide Background Study Report, Holloman AFB, NM (NationView, 2011/NMED 2012). The study reports an upper limit for background lead concentrations of 10.9 mg/kg.

Project Organization

This UFP-QAPP is organized into thirty-seven worksheets as specified by Uniform Federal Policy for QAPPs (AFCEE, 2006). The Policy is intended to insure in an orderly fashion, the problem definition, approach to resolving the problem, quality assurance/quality control activities to insure that the data collected is usable. The table of contents for this document presents a listing of all the UFP-QAPP Worksheets. The field sampling and reporting portions of the UFP-QAPP will be implemented by qualified FPM staff as identified in UFP-QAPP Worksheets #5, 6 and 7. The laboratory analyses will be conducted by Accutest Laboratories Inc. according to DoD Quality Systems Manual (QSM) Version 4.2, October 2010 (UFP-QAPP Worksheet #23).

Scope of Work / Sampling and Analysis

The specific objective of the RI is to further research and confirm the extent of the residual contamination present at the SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range. Furthermore, an investigation to delineate the nature and extent of MC related metals (antimony, arsenic, copper, lead, and zinc) and PAH contamination in soil will be conducted to support, if warranted, a non-time critical removal action. Specifically, the RI scope of work is as follows:

- Review of available background data concerning historical site activities and investigations.
- Based on the results of the previous XRF soil analysis, collect up to an estimated 20 soil samples at each site from shallow soil borings (up to an estimated depth of 3 feet bgs). Analyze for metals (antimony, arsenic, copper, lead, and zinc) using an off-site fixed base laboratory by USEPA SW-846 Method 6010C and for PAHs by USEPA SW-846 Method 8270D Selective Ion Monitoring (SIM). Certain metals are related to specific types of range related activities. For example, arsenic is used as a hardener in shotgun shells, where antimony and lead, but not copper and zinc, may also be associated with shot. However, a uniform analytical approach with respect to these metals will be utilized to account for all potential range related use.
- Analyze up to 10 soil samples per site for Synthetic Precipitation Leaching Procedure (SPLP) analysis (USEPA SPLP SW-846 Method 1312) to obtain additional data to evaluate the soil to groundwater pathway and 5 soils samples per site for Toxicity Characteristic Leaching Procedure (TCLP) analysis for disposal characteristics (USEPA SW-846 Method 1311).
- Compare the validated results to USEPA RSLs (USEPA, November 2013) and NMED soil screening levels to determine the extent of contaminated soils.

- Prepare RI report detailing the results/findings of the RI, which would be used to support a non-time critical removal action if necessary.

Project Records and Data Management Plan

FPM will maintain field records sufficient to recreate all sampling and field measurement activities and to meet all Environmental Restoration Program Information Management System (ERPIMS) data loading requirements. The requirements listed in UFP-QAPP Worksheet #18 will apply to all measuring and sampling activities.

Investigation and Interim Measure Soil Removal Waste Management

Investigation and Interim Measure Soil Removal Waste Management is outlined in QAPP Worksheet #14. Described activities will be completed in accordance to the Project Health and Safety Plan (HSP) (FPM, 2013).

Introduction

The primary purpose of this WP is present the activities and field sampling plan for the RI at the SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range (**Figure 1**). This RI WP is prepared using the UFP-QAPP format http://www.epa.gov/fedfac/pdf/ufp_qapp_worksheets.pdf. The scope of work to be completed for this project is summarized in **Table 1**.

Table 1
Holloman AFB PBR
SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range
Scope of Work

Work Element	Monitoring Matrix	Preliminary Site Chemical of Concern	Site Objective
RI – Field Activities (Sampling)	Soil	MC Metals and PAHs	Site Closeout

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QAPP Worksheet #1 & #2 – Title and Approval Page

1. Project Identifying Information

    a. Site name/project name: Holloman AFB/PBR Remedial Investigation

    b. Site location/number:

    SR859a – Former Skeet Range
    TS862a – Jeep Target Area Skeet Range

    c. Contract/work assignment number: FA8903-13-C-0008; Task Order (TO): NA

2. Lead Organization

    a. Lead Organization Project Manager:

    Laura Peters 2/17/15

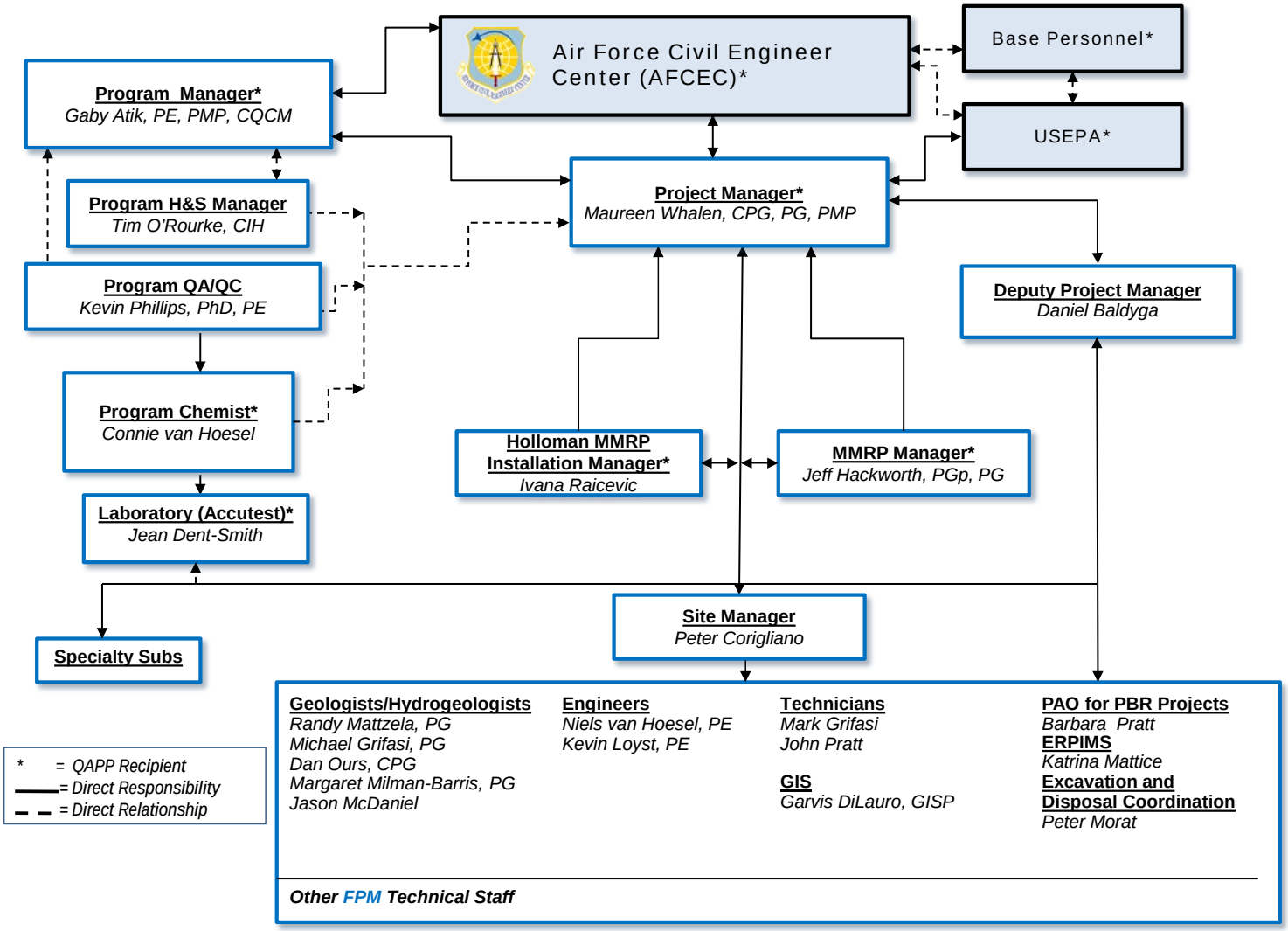
    Laura Peters
    Environmental Restoration Manager
    AFCEC/CZO

    Date

3. Plans and Reports from previous investigations relevant to this project:

Date	Title	Site
2010	Final Report - Modified Comprehensive Site Evaluation Phase I, Holloman Air Force Base, New Mexico	SR859a and TS862a
2011	Holloman Air Force Base, New Mexico – Comprehensive Site Evaluation Phase II Final Work Plan, Military Munitions Response Program.	SR859a and TS862a
2011	Basewide Background Study Report, Holloman Air Force Base, NM.	SR859a and TS862a
2012	Draft Report – Comprehensive Site Evaluation Phase II, Holloman Air Force Base, NM.	SR859a and TS862a
2013	Final Report - Comprehensive Site Evaluation Phase II, Holloman Air Force Base, NM.	SR859a and TS862a

QAPP Worksheet #3 & #5 – Project Organization and QAPP Distribution



QAPP recipients' contact information is provided below:

QAPP Recipients	Title	Organization / Address	Telephone No.	E-mail Address
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Brian Renaghan	Contracting Officer's Representative (COR)	AFCEC/Environmental Center of Excellence Restoration – Execution (CZRX)/2261 Hughes Ave., Suite 155, (JBSA Lackland), Texas 78236-9856	210-395-8581	Brian.renaghan@us.af.mil
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Rita Leal	Contract Specialist	772 ESS/PKB/2261 Hughes Ave., Suite 163, (JBSA Lackland), Texas 78236-9856	210-395-8228	Rita.leal@us.af.mil
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Laura Peters	AFCEC/CZO Environmental Restoration Manager	AFCEC/CZO Bldg. 355, 506 N. Air Commando Way Cannon AFB, NM 88103	575-784-0491	Laura.peters.4@us.af.mil

QAPP Recipients	Title	Organization / Address	Telephone No.	E-mail Address
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Gaby Atik PE	Program Manager	FPM/584 Phoenix Drive, Rome NY, 13441	315-336-7721	g.atik@fpm-remediations.com
Maureen Whalen PG, CPG, PMP	PM	FPM/584 Phoenix Drive, Rome, NY 13441	315-336-7721	m.whalen@fpm-remediations.com
Jeff Hackworth PG	MMRP Manager	FPM/5811 University Heights BLVD., Suite 101, San Antonio, TX 78249	210-495-7744	j.hackworth@fpm-remediations.com
Ivana Raicevic, PhD.	Holloman AFB MMRP Installation Manager	FPM/5811 University Heights BLVD., Suite 101, San Antonio, TX 78249	210-495-7744	i.raicevic@fpm-remediations.com
Jean Dent-Smith	Accutest Laboratories Project Manager	4405 Vineland Road Suite C-15 Orlando, FL 32811	407-425-6700	jeans@accutest.com

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QAPP Worksheet #4, #7, & #8 – Personnel Qualifications and Sign-Off Sheet

Organization: Holloman AFB

Project Personnel	Title	Education/Experience	Specialized Training/Certifications	Signature/Date
Laura Peters	Environmental Restoration Manager			2/17/15

Organization: FPM

Project Personnel	Title	Education/Experience	Specialized Training/Certifications	Signature/Date
Gaby Atik PE	Program Manager	B.S.C.E., Master in Engineering. Engineering Management / Environmental Systems, 20+ years of environmental experience.		2/2/15
Kevin Phillips Ph.D., PE	QA/QC Officer	Ph.D. Environmental Engineering, M.S. Hydrodynamics, B.C.E., Civil Engineering. 30+ years of experience	Licensed Professional Engineer in New York, New Jersey, Pennsylvania, Connecticut, Maryland, Massachusetts, Alabama, and Texas	Kevin J Phillips Digitally signed by Kevin J Phillips DN: cn=Kevin J Phillips, o=FPM Remediation Inc., ou, email=K.phillips@fpm-remediation.com, c=US Date: 2015.02.03 15:48:22 -05'00'
Maureen Whalen PG, CPG, PMP	PM	M.S. Quaternary Studies, B.S. Geology. CPG, PG, PMP. 20 + years of environmental experience	Registered Professional Geologist, Maine, South Carolina	2/2/15
Jeff Hackworth, PGp, PG	MMRP PM	B.S. Geophysics PG 26+ years of environmental experience	Registered Geophysicist: California Registered Professional Geologist: Tennessee	2/2/15
Tim O'Rourke, CIH	Health and Safety (H&S) Manager	Certified Industrial Hygienist 15 years experience		2/2/15

Project Personnel	Title	Education/Experience	Specialized Training/Certifications	Signature/Date
Connie van Hoesel	Program Chemist/Chemical Quality Control (QC) Manager	M.S. Environmental Engineering, B.A. Chemistry, 12 years experience		Concordia van Hoesel 2/2/15
Remedial Investigation Field Team [1]	Remedial Investigation Field Team Personnel	Various	H&S Training per 29 Code of Federal Regulations (CFR) 1910.120 Tailgate meeting to discuss daily plans and procedures	

Organization: Laboratory

Project Personnel	Title	Education/Experience	Specialized Training/Certifications	Signature/Date
Jean Dent-Smith	Accutest Laboratory Inc. PM		Accutest Laboratory is a DoD Accredited laboratory. Accreditation certificate is provided in Appendix B.	Jean Dent-Smith 2/10/15

1 All sampling personnel will be trained using sampling techniques described in the Standard Operating Procedures (SOPs) (Appendix A). All field personnel (including sub-contractors) certifications will be electronically retained by FPM for review.

QAPP Worksheet #6 – Communication Pathways

Communication Drivers	Organization	Name	Contact Info	Procedure (timing, pathways, etc.)
Point of contact (POC) with USEPA Region 6	Holloman AFB RPM	DeAnna Rothhaupt	575-572-3931	Ms. Rothhaupt is the Holloman AFB RPM.
	Environmental Restoration Manager	Laura Peters	575-784-0490	Ms. Peters is the ERM. Primary interface with USEPA Region 6 and ensures projects meet USEPA requirements.
	AFCEC/CZRX - COR	Brian Renaghan	210-395-8633	Mr. Renaghan is the AFCEC COR PM.
	AFCEC/CZRX – COR/Alternative	Layi Oyelowo	210-395-8567	Mr. Oyelowo is the alternate POC to Brian Renaghan.
Overall Project Management	AFCEC/CZRX - COR	Brian Renaghan	210-395-8633	Mr. Renaghan is the AFCEC COR PM.
Program and Project Activities and Issues	FPM Program Manager	Gaby Atik PE	315-336-7721	Is the primary interface with AFCEC and ensures performance objectives are met.
Manages Project; Project activities; Work Plan and/or QAPP Changes	FPM Project Manager/Holloman AFB	Maureen Whalen PG, CPG, PMP	315-336-7721	Overall responsibility of the project. Supervises field sampling activities. Reports to AFCEC and Holloman AFB within three days of the change. Once approved, the UFP-QAPP recipients will receive a copy of the change.
Daily Field Progress Reports	Holloman AFB MMRP Installation Manager	Ivana Raicevic, PhD	210-495-7744	Supervises field sampling and O&M activities. Authors status and completion reports. Reports to Project Manager and/or AFCEC and Holloman AFB within three days of the change. Once approved, the UFP-QAPP recipients will receive a copy of the change.
Sampling and Remediation Activities	Holloman AFB MMRP Installation Manager	Ivana Raicevic, PhD	210-495-7744	Responsible for all sampling and remediation activities to assure goals are attained. Reports to AFCEC and Holloman AFB daily during field efforts.

Communication Drivers	Responsible Entity	Name	Phone No.	Procedure (timing, pathways, etc.)
Project Quality Control/Quality Assurance	QA/QC Officer	Kevin Phillips PhD., PE	631-737-6200	Will determine corrective action for field, data interpretation, and reporting issues
Reporting Lab Data Quality Issues	Chemical Quality Control Manager	Connie van Hoesel	315-336-7721	Will determine corrective action for lab data quality issues
Field and Analytical CAs	Chemical Quality Control Manager	Connie van Hoesel	315-336-7721	Will determine corrective action for field and analytical issues
Release of Analytical Data	Chemical Quality Control Manager	Connie van Hoesel	315-336-7721	No analytical data can be released until it has been reviewed by the Chemical Quality Control Manager and data validation has been completed.
QAPP Amendments	AFCEC/CZRX - COR	Brian Renaghan	210-395-8633	Any major changes to the QAPP must be approved by Brian Renaghan before the changes can be implemented.

QAPP Worksheet #9 – Project Planning Session Summary

No site-specific planning sessions have been held to date, however one will be planned two weeks after the document is submitted for review. Two meetings have been held discussing PBR contract objectives. If any meetings are held with USEPA Region 6, AFCEC, and/or Holloman AFB regarding scoping and/or elements specifically relating to this UFP-QAPP, this worksheet will be revised accordingly.

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QAPP Worksheet #10 – Conceptual Site Model

Site Background

SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range were identified during previous investigations as containing potential sources for munitions MC contamination based on previous range related use. The Former Skeet Range 2 consists of approximately 8 acres of desert scrubland in the southern portion of the installation, north of Forty Niner Avenue and the Holloman runway control tower. The CSE Phase II report recommended splitting the former SR859 MRA into two MRSs: SR859 (34.3 acres) which was recommended for NFA due to lack of MEC and lack of MC exceeding USEPA screening levels and SR859a which was recommended for future munitions response actions. Similarly, the CSE Phase II report recommended splitting the TS862 MRA into two smaller MRSs: TS862 (34.6 acres) which was recommended for NFA due to lack of MEC and lack of MC exceeding USEPA residential RSLs and TS862a which was recommended for future munitions response actions. The TS862a Jeep Target Area Skeet Range occupies approximately 6 acres of desert scrubland in the southeastern portion of the installation just north of the Jeep Target Area.

CSE Phase II XRF soil screening results for lead indicated one exceedance (463 mg/kg) at SR859a Former Skeet Range 2 and no exceedances of the USEPA RSLs (400 mg/kg) for soils at the TS862a Jeep Target Area Skeet Range. Results from fixed base laboratory samples analyzed for PAHs during the CSE Phase II indicated concentrations in soil samples exceeded screening levels in 15 of the 24 samples analyzed at SR859a Former Skeet Range 2 and at least one PAH analyte exceeded screening levels in 16 of 41 samples collected at TS862a Jeep Target Area Skeet Range. CSE Phase II PAH analytical results are provided in **Appendix E**. The results of the CSE Phase II Human Health Risk Screening indicated potential risk to future residential receptors from PAHs in soil.

Previous Investigations

Prior to the modified CSE Phase I there had been no munitions, MEC, or other environmental response activities at the SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range sites.

Modified Comprehensive Site Evaluation Phase I - 2010 – A Modified CSE Phase I was completed in 2010 to characterize the site; evaluate actual or potential releases of hazardous substances, pollutants, or contaminants to migration/exposure pathways (groundwater, soil, and air) from munitions response areas and evaluate associated targets of concern. The Modified CSE Phase I activities compiled and evaluated information related to past military munitions activities, physical site conditions, and future land uses and activities. Information sources included national, regional, and local archives, along with interviews, and observations made during field reconnaissance. No laboratory samples were collected as part of the Modified CSE Phase I activities. Results indicated that the SR859 Former Skeet Range 2 and TS862 Jeep Target Area Skeet Range were utilized as small arms training areas and potential munitions could include expended small arms. Further investigations were warranted to asses if lead and PAHs had been released to the environment. A CSE Phase II was recommended.

Comprehensive Site Evaluation Phase II - 2013 – A CSE Phase II was completed in 2013, and involved the compilation and evaluation of information relating to the possible contamination of environmental media from MC and/or COPC related to historic munitions related activities at

identified MRAs at Holloman AFB. Activities included visual reconnaissance surveys and the collection of soil samples for X-ray fluorescence (XRF) analysis on site for lead and also fixed base laboratory samples for PAHs.

SR859 Former Skeet Range 2 – During CSE Phase II field activities small arms debris associated with 12-gauge shotgun shells, 5.56mm, 7.62mm, and .50-caliber were observed throughout the MRA. Lead shot pellets and dense clay target debris were also scattered throughout the MRA. A total of 140 surface soil samples and 3 subsurface soil samples were collected and analyzed for lead using an XRF analyzer. Detected results ranged from < Limit of Detection (LOD) (12 mg/kg) to 463 mg/kg in surface soil and from below LOD to 71 mg/kg in subsurface soil. Of the 140 surface soil samples 30 were below the LOD of 12 mg/kg. One surface soil sample exceeded the USEPA RSL of 400 mg/kg. Twenty surface and 4 subsurface soil samples were collected and analyzed for PAHs. At least one analyte exceeded the associated human health screening level in 14 of the 24 samples collected. As previously mentioned the CSE Phase II recommended splitting the MRA into two MRSs: SR859 (34.3 acres) was recommended for NFA due to lack of MEC and MC exceeding EPA RSLs and SR859a (8.1 acres) was recommended for further response actions due to range related debris on the surface, and lead and incidental constituents in surface soil that pose potential risk to human health and the environment (USACE, 2013).

TS862a Jeep Target Area Skeet Range – Clay Target debris along with 12-gauge shotgun shells, .45-caliber, and .50-caliber rounds were observed during the CSE Phase II visual reconnaissance surveys. A total of 137 surface soil samples were collected and analyzed for lead using the XRF. Lead analysis results ranged from below the LOD (12mg/kg) to 101 mg/kg. No samples exceeded the screening level of 400 mg/kg, furthermore, 121 samples were below the LOD. Thirty-one (31) surface soil samples and 10 subsurface soil samples were collected for laboratory PAH analysis. Results exceeded the USEPA human health screening levels of at least on analyte in 16 of the 41 soil samples. As previously mentioned the CSE Phase II recommended splitting the MRA into two MRSs: TS862 (34.6 acres) was recommended for NFA due to lack of MEC and MC exceeding EPA RSLs and TS862a (5.7 acres) was recommended for further response actions due to range related debris on the surface, and incidental constituents in surface soil that pose potential risk to human health and the environment (USACE, 2013).

Site Models

The SR859a Former Skeet Range 2 consists primarily of desert scrubland and is not managed for ecological habitat. One XRF screening samples exceeded the USEPA RSL of 400 mg/kg and 14 of 24 soil samples indicated at least one PAH concentration in soil exceeding screening levels. Complete pathways were identified through the food chain exposure for biota from vegetation and wildlife and also through surface and subsurface soil by ingestion, dermal contact, and inhalation for base personnel, current and future contractors, future recreational users, future residents, and biota.

The TS862a Jeep Target Area Skeet Range consists primarily of desert scrubland and is not managed for ecological habitat. No XRF screening samples exceeded the USEPA RSL of 400 mg/kg. At least one PAH analyte exceeded human health screening levels in 16 of the 41 samples analyzed. The CSE Phase II identified complete pathways through the food chain exposure for biota from vegetation and wildlife and also through surface soil by ingestion, dermal contact, and inhalation for all receptors.

There is also a potential for residual on-site surface soil containing metals contamination and PAHs to be transported from the site via wind erosion.

Access to Holloman AFB requires admittance through a security gate, and there is a fence around the perimeter of the installation. There is restricted access controls associated with theSR859a Former Skeet Range 2 due to its location within the flight line. Access is limited to authorized personnel and contractors. Flight line driver training or an escort are required. Trespasser access to the flight line is unlikely. TS862a Jeep Target Area Skeet Range is accessed through locked gates however, the site may be accessed by a heavily rutted dirt road bypassing any gates.

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QAPP Worksheet #11 – Project/Data Quality Objectives

1. State the Problem

What are the nature and extent of metals (antimony, arsenic, copper, lead, and zinc) and PAH contamination in the soil at the SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range sites? Is further action necessary to address residual concentrations of metals (antimony, arsenic, copper, lead, and zinc) and PAHs that may pose a threat to human health or is no further action (NFA) appropriate?

2. Identify the Goals of the Study

The specific objective of this investigation is to develop a refined and updated understanding of the extent (vertical and horizontal) and distribution of metals and PAHs in soil. This information will be used to support a non-time critical removal action at each former range if necessary. It should be noted that certain metals are related to specific types of range related activities. For example, arsenic is used as a hardener in shotgun shells, where antimony and lead, but not copper and zinc, may also be associated with shot. However, a uniform analytical approach with respect to these metals will be utilized to cover all potential range related use.

3. Identify Information Inputs

Soil samples will be collected (locations will be based on previous investigations) and soil samples will be analyzed at an off-site laboratory (Accutest Laboratories, Inc.) for metals (antimony, arsenic, copper, lead, and zinc) (by USEPA SW-846 Method 6010C) and PAHs (USEPA SW-846 Method 8270D). If necessary (i.e., the screening and/or analytical result indicates concentrations of metals or PAHs present in soil exceed the project action limits) (see Worksheet #15) the sample will be analyzed using USEPA's SPLP (USEPA SPLP SW-846 Method 1312) to determine if the analyte will leach from the soil.

4. Define the Boundaries of the Study

The spatial limits of work at each MRS are illustrated in **Figures 2** through **5.** Sampling locations (surface soils and subsurface to 3ft bgs) for metals (antimony, arsenic, copper, lead, and zinc) and PAHs are shown in **Figures 4** and **5**. Certain metals are related to specific types of range related activities. For example, arsenic is used as a hardener in shotgun shells, where antimony and lead, but not copper and zinc, may also be associated with shot. However, a uniform analytical approach with respect to these metals will be utilized to account for all potential range related use. Residual PAH compounds in soil are likely a result from the breakdown of clay targets.

5. Develop the Analytical Approach

The following decision rules will be utilized to link potential results with conclusions or future actions.

Decision Rule 1. Soil samples from 0 to 3ft bgs will be collected for metals (antimony, arsenic, copper, lead, and zinc) and PAH analysis to determine the presence of contamination, as follows:

- ***If*** metals and/or PAHs are detected above the LOD (see Worksheet #15) in a sample, ***then*** it will be determined that the corresponding compound is present and included as a detect in the existing analytical dataset for the site (see Decision Rule 2).

- ***If*** metals and/or PAHs are not detected above the LOD (see Worksheet #15) in a sample, ***then*** it will be determined that the compound is not present and included as a non-detect in the existing analytical dataset for the site (see Decision Rule 2).

Decision Rule 2. The site will be reassessed based on the existing dataset (comprised of historical and new data) to determine if further action is necessary, as follows:

- ***If*** the concentrations of metals and/or PAHs in soil are less than the respective project action limits (see Worksheet #15), ***then*** it will be determined that no further remedial action is accepted.
- ***If*** the concentrations of metals and/or PAHs in soil exceed the project action limits (see Worksheet #15), ***then*** a range of data points exceeding the project action limits from the site dataset will be analyzed to determine if the analytes will leach from the soil utilizing USEPA's synthetic precipitation leaching procedure (SPLP) (USEPA SPLP SW-846 Method 1312) (see Decision Rule 3).

Decision Rule 3. The site will be further assessed based on the current data set to determine if further action is necessary, as follows:

- ***If*** the leachate concentrations in soil are below the respective project action limits (see Worksheet #15), ***then*** it will be determined that no further remedial action is accepted.
- ***If*** the leachate concentrations present in soil are greater than the respective project action limits (see Worksheet #15), ***then*** it will be determined that soil removal may be required of the soil that will result in a groundwater concentration that exceeds the project action limits (see Worksheet #15) (see Decision Rule 4).

Decision Rule 4. Removal of contaminated soil that exceeds the project action limits (see Worksheet #15) with confirmation soil sampling data to determine if further action is necessary, as follows:

- ***If*** the excavation confirmation concentrations of metals and/or PAHs present in soil are less than the respective project action limits (see Worksheet #15), ***then*** it will be determined that no further remedial action is accepted.
- ***If*** the excavation confirmation concentrations of metals and/or PAHs present in soil are greater than the respective project action limits (see Worksheet #15), ***then*** it will be determined that removal will be required of the material that exceeds the project action limits (see Worksheet #15) (repeat Decision Rule 4).

6. Specify Performance or Acceptance Criteria

Sample analytical results will be compared to the project action limits as shown in Worksheet #15. Worksheet #37 describes the usability assessment of the data. Decision errors include data quality and usability. To ensure the quality of the data, all data will be reviewed, verified, and validated in accordance with this QAPP. To ensure usability of laboratory data, appropriate laboratory methods have been selected to provide the necessary laboratory detection limits (DLs). Acceptance criteria for the analytical data are listed in Worksheet #28.

7. Develop the Detailed Plan for Obtaining Data

The sampling design and rationale are presented in Worksheet #17. Worksheets #16, #17, and #18 describe the details of the sampling. Worksheets #19, #20, #24-28, and #30 will specify analysis design requirements.

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QAPP Worksheet #12 – Measurement Performance Criteria

Matrix	**Soil**	**Data qualifier definitions, and full data review/validation criteria are listed in Tables 12-2 through 12-4.**			
Analytical Group	**Metals**				
Conc. Level	**Low to High**				
Sampling Procedure[1]	**Analytical Method/ Standard Operating Procedure (SOP)[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOPs No. 1, and No. 2	EPA SW-846 6010C / SOP MET100 USEPA SPLP 1312/SOP OP042/	Precision – Lab	Relative Percent Difference (RPD) <10%	Matrix spike/Matrix spike duplicate (MS/MSD) and/or Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD) Proficiency Testing (PT) Sample	A
			Refer to Worksheet #24	Calibration – Initial and Continuing	
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x limit of quantification (LOQ), then 20% RPD for aqueous samples, 30% for soil. If either the parent or duplicate value is < 5X the LOQ, then the difference between the parent and duplicate must be < 2X the LOQ.	Field Duplicates	S&A
		Accuracy/Bias	See Table 12-1	Interference Check Sample, LCS, MS, and post digestion spike (PDS), if applicable	A
			Refer to Worksheet #24	Calibration – Initial and Continuing	

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOPs No. 1 and No. 2 (continued)	EPA SW-846 6010C / SOP MET100 USEPA SPLP 1312/SOP OP042	Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A
				Equipment blanks	S&A
		Representativeness	Holding time compliance per 40 CFR 136 and/or method	Holding time	A
		Quantitation Limit	LOQ > LOD LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ as the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the DL.	Sample results that are less than the LOQ, but greater than the DL, will be reported with a **J**-flag. Quarterly LOD verification.	A
		Completeness	90 and 95% for soil	Data Completeness Check	S&A

[1]Reference No. from QAPP Worksheet #21

[2]Reference No. from QAPP Worksheet #23

QAPP Worksheet #12 – Measurement Performance Criteria

Matrix	**Soil**	**Data qualifier definitions, and full data review/validation criteria are listed in Tables 12-2 through 12-4**			
Analytical Group	**PAHs**				
Conc. Level	**Low to High**				
Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), and/or Analytical (A)**
SOPs No. 1 and No. 2	USEPA 8270D by Selected Ion Monitoring (SIM)/SOP MS008 USEPA SPLP 1312/SOP OP042	Precision – Lab	RPD <30%	MS/MSD, LCS/LCSD, and/or PT Sample	A
			Refer to Worksheet #24	Calibration – Initial and Continuing	
		Precision – Field/Laboratory	If both the parent and duplicate values are > 5x limit of quantification (LOQ), then 20% RPD for aqueous samples, 30% for soil. If either the parent or duplicate value is < 5X the LOQ, then the difference between the parent and duplicate must be < 2X the LOQ.	Field Duplicates	S&A
		Accuracy/Bias	See Table 12-1	Interference Check Sample, LCS, MS, and post digestion spike (PDS), if applicable	A
			Refer to Worksheet #24	Calibration – Initial and Continuing	

QAPP Worksheet #12 – Measurement Performance Criteria

Sampling Procedure[1]	Analytical Method/ SOP[2]	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), and/or Analytical (A)
SOPs No. 1 and No. 2 (continued)	USEPA 8270D by Selected Ion Monitoring (SIM)/SOP MS008 USEPA SPLP 1312/SOP OP042	Accuracy/Bias Contamination	No target compounds > ½ LOQ	Method blanks	A
				Equipment blanks	S&A
		Representativeness	Holding time compliance per 40 CFR 136 and/or method	Holding time	A
		Quantitation Limit	LOQ > LOD LOD & LOQ must be verified quarterly.	Standard that is at or below the LOQ as the lowest point on the calibration curve.	A
		Sensitivity	Sample results will be reported to the DL.	Sample results that are less than the LOQ, but greater than the DL, will be reported with a **J**-flag. Quarterly LOD verification.	A
		Completeness	90 and 95% for soil	Data Completeness Check	S&A

[1]Reference No. from QAPP Worksheet #21

[2]Reference No. from QAPP Worksheet #23

Table 12-1
Accuracy and Precision Criteria for Chemical Analysis

Spiking Compound	Accuracy (%R)				Precision (RPD)		
	Aqueous		Soil		Aqueous		Soil
METALS							
Antimony	80-120		80-120		20		20
Arsenic	80-120		80-120		20		20
Copper	80-120		80-120		20		20
Lead	80-120		80-120		20		30
Zinc	80-120		80-120		20		20
PAHs							
Acenapthene	45 - 110		45 - 110		30		30
Acenaphthylene	50 - 105		45 - 105		30		30
Benzo(a)anthracene	55 - 110		50 - 110		30		30
Benzo(a)pyrene	55 - 110		50 - 110		30		30
Benzo(b)fluoranthene	45 - 120		45 - 115		30		30
Benzo(g,h,i)perylene	40 - 125		40 - 125		30		30
Benzo(k)fluoranthene	45 - 125		45 - 125		30		30
Chrysene	55 - 110		55 - 110		30		30
Dibenz(a,h)anthracene	40 - 125		40 - 125		30		30
Fluoranthene	55 - 115		55 - 115		30		30
Fluorene	50 - 110		50 - 110		30		30
Indeno(1,2,3-cd)pyrene	45 - 125		40 - 120		30		30
2-Methylnaphthalene	45 - 105		45 - 105		30		30
Naphthalene	40 - 100		40 - 105		30		30
Phenanthrene	50 - 115		50 - 110		30		30
Pyrene	50 - 130		45 - 125		30		30

Notes:

%R - Percent Recovery

RPD - Relative Percent Difference

Table 12-2
Data Qualifier Definitions

Qualifier	Description
J	Estimated. The analyte was positively identified; the quantitation is an estimation due to discrepancies in meeting certain analyte-specific QC criteria, or the concentration is less than the sample quantitation limit.
UJ	The analyte was not detected. The result is estimated due to discrepancies in meeting certain analyte-specific QC criteria.
B	Blank contamination. The analyte was found in the sample at a concentration similar to that observed in a blank.
R	Rejected. The data are rejected due to deficiencies in meeting QC criteria and may not be used for decision-making.

Table 12-3
Data Review/Validation Criteria for USEPA Method 6010C

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
Limit of Detection (LOD) determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DoD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R**-flag to data without a valid LOD verification
Limit of Quantification (LOQ) establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DoD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	Not available (N/A)	N/A	N/A
Instrument Detection Limit (IDL) study (Inductively coupled plasma [ICP] only)	At initial set-up and after significant change	Detection limits established shall be ≤ LOD	N/A	Samples cannot be analyzed without a IDL.	Apply **R**-flag to data without a valid IDL study
Linear dynamic range or high-level check standard (ICP only)	Every 6 months	Within ±10% of true value	N/A	N/A	N/A
Holding time	Every sample	Soil samples: 6 months	Contact FPM as to additional measures to be taken.	N/A	Apply **J**-flag to detects and **UJ**-flag to non-detects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to non-detects to samples > 2X holding time criteria.
Sample temperature	None	N/A	Contact FPM for additional measures to be taken.	N/A	N/A
Initial calibration for all analytes (ICAL)	Daily initial calibration prior to sample analysis	$r \geq 0.995$	Correct problem then repeat initial calibration	Problem must be corrected. No samples may be run until ICAL has passed.	Apply **R**-flag to data without a valid ICAL
ICP: minimum one high standard and a blank					

Second source calibration verification (ICV)		Once after each initial calibration, prior to sample analysis		Value of second source for all analytes within ± 10% of expected value (initial source)		Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.		Problem must be corrected. No samples may be run until calibration has been verified.		Apply **R**-flag to data without second source verification
Continuing Calibration verification (CCV)		After every 10 samples and at the end of the analysis sequence.		ICP: All analytes within ± 10% of expected value from ICAL.		Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.		Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.		Apply **R**-flag to data with CCV outside criteria.
Low-level calibration check standard		Daily, after one-point ICAL.		Within + 20% of true value.		Correct problem, then reanalyze.		Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.		Apply **R**-flag to data with low-level calibration check standard outside criteria.
Method blank, Equipment blank		One per preparatory batch, one per sampling day		No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL and > 1/10 the amount measured in any sample or 1/10 the regulat		Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.		Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.		Apply **B**-flag to analytes detected in field samples < 5X blank contamination.
Calibration blank		Before beginning a sample run, after every 10 samples, and at the end of the analysis sequence		No analytes detected > LOD		Correct problem. If required, reprep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.		Flagging is only appropriate in cases where the samples cannot be reanalyzed.		Apply **B**-flag to analytes detected in field samples < 5X blank contamination.

Interference check solutions (ICS) (ICP only)		At the beginning of an analytical run		ICS-A: Absolute value of concentration for all nonspiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes). ICS-AB: Within ± 20% of expected value.		Terminate analysis; locate and correct problem; reanalyze ICS.		No samples may be analyzed without a valid ICS		Apply **R**-flag to data with ICS outside criteria.
Laboratory control sample (LCS)		One per preparatory batch		QC acceptance criteria specified in UFP-QAPP Table 12-1.		Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.		Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.		High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to non-detects. Very low bias (ICP Metals %R<60%, Hg %R < 50%): Apply **J**-flag to detects and **R**-flag to non-detects.
Dilution test (ICP only)		Each preparatory batch or when a new or unusual matrix is encountered		Five fold dilution must agree within ± 10% of the original determination.		ICP: Perform post-digestion spike (PDS) addition.		Only applicable for samples with concentrations > 50X LOQ (6010B)		Apply **J**-flag to analytes in parent sample outside criteria
PDS (ICP only)		When dilution test fails or analyte concentration in all samples < 50X LOD		75-125%		See flagging criteria.		The spike addition should produce a level between 10-100X LOQ		Apply **J**-flag to analytes in parent sample outside criteria
Matrix spike/Matrix spike duplicate (MS/MSD)		One per preparatory batch per matrix		QC acceptance criteria specified in UFP-QAPP Table 12-1.		Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.		For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. No data flagging if native concentrations are > 4X spiking amount		For the specific analyte(s) in the parent sample, apply **M**-flag to detects if acceptance criteria are not met. MS/MSD data should not be used alone to qualify data.
Laboratory sample duplicate		One per preparatory batch per matrix (if MS/MSD is not performed)		RPD ≤ 20% (sample and sample duplicate)		Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.		Data shall be evaluated to determine the source of difference.		For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.
Field Duplicate		One per 10 field samples		See UFP-QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2)		N/A		N/A		Apply **J**-flag to detects and **UJ**-flag to non-detects.
Results reported between DL and LOQ		N/A		N/A		N/A		N/A		Appy **J**-flag to all results between DL and LOQ.

Table 12-4
Data Review/Validation Criteria for USEPA Method 8270D SIM

QC Check	Minimum Frequency	Acceptance Criteria	Laboratory Corrective Action	Comments	FPM Flagging Criteria
LOD determination and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOD verification checks shall be performed.	See DoD QSM v 4.2. LOD verification checks must produce a signal at least 3 times the instrument's noise level.	Repeat detection limit determination and LOD verification check at higher level and set LOD.	LOD is 2-3x the detection limit (for a single-analyte standard) or greater than 1-4x the detection limit (for a multi-analyte standard).	Apply **R**-flag to data without a valid LOD verification
LOQ establishment and verification	At initial set-up and subsequently once per 12-month period; otherwise quarterly LOQ verification checks shall be performed.	See DoD QSM v 4.2. LOQ must be set within the calibration range prior to sample analysis.	N/A	N/A	N/A
Holding time	Every sample	Soil VOCs: 48 hours until frozen by laboratory (< -7°C), 14 days to analysis. Soil SVOCs: 14 days to extract, 40 days to analysis.	Contact FPM as to additional measures to be taken.	None	Apply **J**-flag to detects and **UJ**-flag to non-detects to samples < 2X holding time criteria. Apply **J**-flag to detects and **R**-flag to non-detects to samples > 2X holding time criteria.
		Water VOCs: 7 days unpreserved, 14 days preserved (pH<2, HCl) Groundwater SVOCs: 7 days to extract, 40 days to analysis.			
Sample temperature	Every cooler	4±2 °C	Contact FPM as to additional measures to be taken.	None	Samples arriving at temperature 6-10°C, apply **J**-flag to detects and **UJ**-flag to non-detects.
					VOC samples received at temperature > 10°C, **R**-flag all results.
Sample preservation	Every sample	Sample preservation requirements not met (e.g., pH >2, headspace in VOA vials, etc.)	Contact FPM as to additional measures to be taken.	None	Apply **J**-flag to detects and **UJ**-flag to non-detects.
Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Problem must be corrected. No samples may be accepted without a valid tune.	Apply **R**-flag to data without a valid tune

Breakdown check (DDT method 8270C only)		At the beginning of each 12-hour period, prior to analysis of samples		Degradation ≤ 20% for DDT.		Correct problem then repeat breakdown check				Apply **R**-flag to data analyzed if DDT degradation is not met.
Minimum five point initial calibration for all analytes (ICAL)		ICAL prior to sample analysis		1. Average response factor (RF) for SPCCs: VOCs - ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050		Correct problem then repeat initial calibration		Problem must be corrected. No samples may be run until ICAL has passed		Apply **R**-flag to data without a valid ICAL
				2. RSD for RFs for CCCs: VOCs and SVOCs ≤ 30% and one option below;						Apply **R**-flag to data without a valid ICAL
				Option 1: RSD for each analyte ≤ 15%						
				Option 2: linear least squares regression r ≥ 0.995						
				Option 3: non-linear regression - coefficient of determination (COD) r^2 ≥ 0.99 (6 points shall be used for second order, 7 points shall be used for third order)						
Second source calibration verification		Once after each ICAL		Value of second source for all analytes within ± 20% of expected value (initial source)		Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.		Problem must be corrected. No samples may be run until calibration has been verified.		Apply **R**-flag to data without second source verification.
Evaluation of relative retention times (RRTs)		Each sample		RRT of each target analyte in each calibration standard within ± 0.06 RRT units.		Correct problem, then rerun ICAL		Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance.		Apply **R**-flag to data outside retention time window
Manual Integration		All		Acceptance by FPM Chemist		Provide justification for each instance of manual integration		Laboratory will provide chromatograms before and after each manual integration		Apply **R**-flag to all compounds with improper integration

Calibration verification (CV)		Daily, before sample analysis, and every 12 hours of analysis time.		Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050						Apply **J**-flag to detects and **UJ**-flag to non-detects if average RF not met
				% Difference/Drift (%D) for all target compounds and surrogates: VOCs and SVOCs ≤ 20%D (Note: D ≤ difference when using RFs or drift when using least squares regression or non-linear calibration.)		Correct problem, then rerun CV. If that fails, repeat ICAL. Reanalyze all samples since last acceptable CCV.		Problem must be corrected. No results may be reported without a valid CCV. Flagging criteria is only appropriate in cases where the samples cannot be reanalyzed.		High bias: Apply **J**-flag to detects Low bias: Apply **J**-flag to detects and **R**-flag to non-detects
Internal standards verification		In all field samples and standards		Retention time ± 30 seconds from retention time of the midpoint standard in the CV		Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.		Sample results are not acceptable without a valid IS verification.		If corrective action fails in field samples, apply **J**-flag to detects and **UJ**-flag to non-detects to analytes with IS recoveries between 30%-50% or > 150%. Apply **R**-flag to samples with IS recoveries < 30%.
				Extracted ion current profile (EICP) area within - 50% to + 100% of ICAL midpoint standard						
Method blank, Equipment blank, Trip blank (VOCs only)		One per preparatory batch, one per sampling day, one per storage cooler		No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit.		Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.		Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.		Apply **B**-flag to analytes detected in field samples < 5X blank contamination (<10X for common laboratory contaminants).
Laboratory control sample (LCS)		One per preparatory batch		QC acceptance criteria specified in UFP-QAPP Table 12-1.		Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.		Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.		High bias: Apply **J**-flag to detects. Low bias: Apply **J**-flag to detects and **UJ**-flag to non-detects. Very low bias (%R<30%): Apply **J**-flag to detects and **R**-flag to non-detects.

Matrix spike/Matrix spike duplicate (MS/MSD)		One per preparatory batch per matrix		QC acceptance criteria specified in UFP-QAPP Tables 12-1.		Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.		For matrix evaluation only. If MS results are outside QC limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.		For the specific analyte(s) in the parent sample, apply **M**-flag to detects if acceptance criteria are not met. MS/MSD data should not be used alone to qualify data.
Laboratory sample duplicate		One per preparatory batch per matrix (if MS/MSD is not performed)		RPD $\leq$ 30% (sample and sample duplicate)		Examine the project-specific DQOs. Contact FPM as to additional measures to be taken.		For the specific analyte(s) in the parent sample, apply **J**-flag to detects if acceptance criteria are not met.		Data shall be evaluated to determine the source of difference. Apply **J**-flag to detects if acceptance criteria are not met.
Surrogate spike		All field and QC samples		QC acceptance criteria specified in UFP-QAPP Table 12-2.		For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available.		Analytes identified in UFP-QAPP Table 12-6.		High bias: Apply **J**-flag to detects Low bias: Apply **J**-flag to detects and **UJ**-flag to non-detects. Very low bias (%R<10%): Apply **J**-flag to detects and **R**-flag to non-detects. For SVOCs, no qualification when only one surrogate is outside QC criteria.
Results reported between DL and LOQ		N/A		N/A		N/A		N/A		Appy **J**-flag to all results between DL and LOQ.
Field Duplicate		One per 10 field samples		See UFP-QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2).		N/A		N/A		Appy **J**-flag to detects and **UJ**-flag to non-detects.

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QAPP Worksheet #13 – Secondary Data Uses and Limitations

Data Type	Data Source	How Data Will Be Used	Limitations on Data Use
Soil Data	Holloman Air Force Base, Basewide Background Study Report NationView, LLC., NMED.	To assess potential areas of contamination and focus data collection activities in specific site areas where contamination is most likely. To determine if further soil sample collection appears warranted or to establish contamination/excavation boundaries.	Secondary data may not meet all DQOs, and, therefore, may not be able to be used without limitation.
Soil Data	Holloman Air Force Base, Comprehensive Site Evaluation Phase II Report, September 2013 (USACE) Historical maps, records, and various documents relating to historic site use, CSE Phase I information, soil sample collection and contamination delineation. USACE and HDR Environmental, Operations and Construction, Inc.		

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QAPP Worksheet #14 and #16 – Project Tasks & Schedule

The project schedule is provided in **Appendix C.**

Summary of Project Tasks

Sampling Tasks (Performed by FPM)**:**

- Specific discussion of the sampling approach and sampling design and rationale is provided in Worksheet #17.
- Soil sampling will assess the absence or presence of contamination. Soil sampling will assess the effectiveness of excavations removing residual contamination. Sample locations will be surveyed using Differential Global Positioning System (DGPS) equipment.
- Samples will be collected using the SOPs attached as Appendix A of this UFP-QAPP.

Analysis Tasks (Performed by Accutest)**:**

- Accutest Laboratories, Inc. will analyze samples for metals using USEPA SW-846 Method 6010C and PAHs using USEPA SW-846 Method 8270D SIM.
- SPLP (USEPA SPLP SW-846 Method 1312) will be analyzed for potential mobility of contamination present in soils from each site.

QC Tasks – All Projects:

1. MS/MSDs will be collected at an approximate frequency of 5%.
2. Duplicates will be collected at a rate of 10% and analyzed by Accutest to assess field and laboratory precision.
3. Equipment blanks will be collected from each type of non-disposable, decontaminated sampling device.
4. Laboratory performance evaluation samples will be collected at each site to assess the laboratory's ability to provide defensible data of a known quality.
5. Data validation will be conducted on 100% of all analytical data collected.

Secondary Data – All Projects:

Previously collected data will be evaluated. Secondary data may not meet all DQOs, and, therefore, may not be able to be used without limitation. See Worksheet #13.

Summary of Project Tasks

Data Management Tasks – All Projects: Data will be delivered in an ERPIMS database compatible format after data verification/ validation have been performed and data qualifiers have been added.
Waste Management Tasks – All Projects: 1. Soils sampled but not used for laboratory analysis will be containerized in 55 gallon Department of Transportation approved drums. Soils contained in 55 gallon drums will be characterized for proper disposal off site. Decontamination water (if non disposable sampling equipment is used) will also be collected in drums and analyzed disposal off site.
Documentation and Records – All Projects: 1. All field documentation will be recorded in indelible ink in bound field books. These will summarize all daily field activities, weather conditions, personnel present, visitors, etc. All samples collected will be documented as to their location, which will be measured using a DGPS. Each day's samples and associated field measurements shall be recorded on field sampling forms. Chain of Custody (CoC) forms, bills of lading, airbills, and sample logs will be prepared and retained for each sample. 2. A copy of the final UFP-QAPP will be retained in a central project file (electronically on a server) and in print form in the onsite office, as well as in the Administrative Record.
Data Packages – All Projects: Accutest Laboratories, Inc. will complete analytical data packages in accordance with the AFCEC approved forms or similar and will provide ERPIMS X file.
Assessment / Audit Tasks – All projects: Field Sample Collection and Documentation Audits: to be determined (TBD)

Summary of Project Tasks

Data Review Tasks – All projects

1. For the samples, Accutest Laboratories, Inc. will verify that all data are complete for samples received. All data package deliverable requirements will be met. Data will be 100% verified by FPM in accordance with this UFP-QAPP. A data verification report will be prepared for each lab work order (lab data package).

2. Verified and validated data and all related field logbooks/notes/records will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined and data will be compared to Project Quality Objectives and required Action Limits. Corrective Action will be completed as necessary. Final validated data are placed in the ERPIMS database, with any necessary qualifiers and tables, charts and graphs generated.

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QAPP Worksheet #15 – Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

Matrix: Soils
Analytical Group: Metals (SW-846 Method 6010C)
Concentration Level: Low to Medium

Analyte	Chemical Abstracts Service (CAS) Number	USEPA Screening Levels[1] Residential Soil (mg/kg)	NMED Hazardous Waste Bureau Residential Soil SSL (mg/kg)[2]	Achievable Laboratory Limits[3]	
				Level of Detection (LOD) (mg/kg)	Limit of Quantitation (LOQ) (mg/kg)
Antimony	7440-36-0	3.1	31.3	0.1	1
Arsenic	7440-38-2	0.67	3.9	0.1	0.5
Copper	7440-50-8	310	3,130	0.1	1.25
Lead	7439-92-1	400	400	0.1	1
Zinc	7440-66-6	2300	23,500	0.25	1

Notes:
NA Not Applicable
(1) May 2014 USEPA Regional Residential Screening Levels.
(2) NMED Soil Screening Levels February 2012 (updated June 2012).
(3) Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.

QAPP Worksheet #15 – Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

Matrix: Soils
Analytical Group: PAHs (SW-846 Method 8270D SIM)
Concentration Level: Low to Medium

Analyte	Chemical Abstracts Service (CAS) Number	USEPA Screening Levels[1] Residential Soil (mg/kg)	NMED Hazardous Waste Bureau Residential Soil SSL (mg/kg)[2]	Achievable Laboratory Limits[3]	
				Level of Detection (LOD) (mg/kg)	Limit of Quantitation (LOQ) (mg/kg)
Acenaphthene	83-32-9	350	3,440	0.067	0.13
Acenaphthylene	208-96-8	NA	NA	0.067	0.13
Benzo(a)anthracene	56-55-3	0.15	1.48	0.013	0.027
Benzo(a)pyrene	50-32-8	0.015	0.48	0.013	0.027
Benzo(b)fluoranthene	205-99-2	0.15	1.48	0.013	0.027
Benzo(g,h,i)perylene	191-24-2	NA	NA	0.013	0.027
Benzo(k)fluoranthene	207-08-9	1.5	14.8	0.013	0.027
Chrysene	218-01-9	15	148	0.013	0.027
Dibenz(a,h)anthracene	53-70-3	0.015	0.148	0.013	0.027
Fluoranthene	206-44-0	230	2,290	0.067	0.130
Fluorene	86-73-7	230	2,290	0.067	0.130
Indeno(1,2,3-cd)pyrene	193-39-5	0.15	1.48	0.013	0.027
2-methylnaphthalene	91-57-6	23	NA	0.067	0.130
Naphthalene	91-20-3	3.8	43.0	0.067	0.130
Phenanthrene	85-01-8	NA	1,830	0.067	0.130
Pyrene	129-00-0	170	1,720	0.067	0.130

Notes: NA Not Applicable
(1) May 2014 USEPA Regional Residential Screening Levels.
(2) NMED Soil Screening Levels February 2012 (updated June 2012).
(3) Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.

QAPP Worksheet #15 – Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

Matrix: Soil, SPLP Leachate
Analytical Group: Metals (SW-846 Method 6010C and SPLP SW-846 Method 1312)
Concentration Level: Low

Analyte	Chemical Abstracts Service (CAS) Number	USEPA Maximum Contaminant Level (µg/L)[1]	NMED Water Quality Standard (µg/L)[2]	Achievable Laboratory Limits[3]	
				Level of Detection (LOD) (µg/L)	Limit of Quantitation (LOQ) (µg/L)
Antimony	7440-36-0	6	NA	2	6
Arsenic	7440-38-2	10	100	5	10
Copper	7440-50-8	1,300	NA	2	25
Lead	7439-92-1	15	50	2	5
Zinc	7440-66-6	NA	NA	5	20

Notes: NA Not Applicable
(1) May 2014 USEPA Regional Residential Screening Levels MCL.
(2) NMED Water Quality Control Commission Groundwater and Surface Water Protection Human Health Standard
(3) Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.

QAPP Worksheet #15 – Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

Matrix: SPLP Leachate
Analytical Group: PAHs (SW-846 Method 8270D SIM and SPLP SW-846 Method 1312)
Concentration Level: Low

Analyte	Chemical Abstracts Service (CAS) Number	USEPA Maximum Contaminant Level (µg/L)[1]	NMED Water Quality Standard (µg/L)[2]	Achievable Laboratory Limits[3]	
				Level of Detection (LOD) (µg/L)	Limit of Quantitation (LOQ) (µg/L)
Acenaphthene	83-32-9	NA	NA	0.5	1
Acenaphthylene	208-96-8	NA	NA	0.5	1
Benzo(a)anthracene	56-55-3	NA	NA	0.05	0.2
Benzo(a)pyrene	50-32-8	0.2	0.7	0.05	0.2
Benzo(b)fluoranthene	205-99-2	NA	NA	0.05	0.2
Benzo(g,h,i)perylene	191-24-2	NA	NA	0.05	0.2
Benzo(k)fluoranthene	207-08-9	NA	NA	0.05	0.2
Chrysene	218-01-9	NA	NA	0.1	0.2
Dibenz(a,h)anthracene	53-70-3	NA	NA	0.05	0.2
Fluoranthene	206-44-0	NA	NA	0.5	1
Fluorene	86-73-7	NA	NA	0.5	1
Indeno(1,2,3-cd)pyrene	193-39-5	NA	NA	0.05	0.2
2-methylnaphthalene	91-57-6	NA	30	0.5	1
Naphthalene	91-20-3	NA	30	0.5	1
Phenanthrene	85-01-8	NA	NA	0.5	1
Pyrene	129-00-0	NA	NA	0.5	1

(1) May 2014 USEPA Regional Residential Screening Levels MCL.
(2) NMED Water Quality Control Commission Groundwater and Surface Water Protection Human Health Standard.
(3) Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.

QAPP Worksheet #17 – Sampling Design and Rationale

The Holloman SR859a Former Skeet Range 2 and TS862a Jeep Target Area Skeet Range locations are shown in **Figure 1**. Site specific details are provided below. All field parameter measurements will be documented in the daily chemical QC reports issued as part of the RI Report.

Soil samples will be collected to further delineate soil contamination above USEPA residential screening levels for metals and PAHs. Soils will also be analyzed using the SPLP analytical methods to determine if the remaining concentrations in soils pose a threat to groundwater. TCLP samples will also be collected and analyzed to determine disposal characterization. Results will be used to determine if additional actions are necessary at the site. Previously collected background soil sample data will be used during data evaluation to determine potentially impacted soil areas. This information will be provided in the RI report.

SR859a Former Skeet Range 2

Building on historical soil sampling and XRF screening results, an estimated 20 shallow soil samples (from 0 ft to an estimated depth of 3 to 5ft bgs) will be collected from previously identified areas to confirm the previously delineated soil contamination above the USEPA residential RSLs for metals (antimony, arsenic, copper, lead, and zinc) and PAHs. Approximately ten (10) soil samples (consisting of a range of samples collected from the data set) will also be analyzed using the SPLP analytical methods to determine if the remaining contaminated soils pose a threat to groundwater. Results will be used to determine remedial options for impacted areas at the site. Remedial options will be evaluated in an Engineering Evaluation/Cost Analysis (EE/CA). Proposed sampling locations with historical results are shown in **Figure 4**.

TS862a Jeep Target Area Skeet Range

Utilizing historical data, approximately 20 shallow soil samples will be collected from areas previously identified as containing clay target debris and areas containing PAH contamination based on skeet range lead shot dispersion. Proposed sample locations and historical data are shown on **Figure 5**. In addition, approximately ten (10) soil samples (consisting of a range of samples collected from the data set) will also be analyzed using the SPLP analytical methods to determine if the remaining contaminated soils pose a threat to groundwater. Results will be used to determine remedial options for impacted areas at the site. Remedial options will be evaluated in an EE/CA.

Soil Sampling

Soil samples will be collected at depth intervals ranging from 0 to 36 inches bgs depending on the location and historical sample data from the adjacent area. Samples will be analyzed for metals by USEPA Method 6010C and PAHs by USEPA method 8270D SIM. Results from laboratory analysis will be used to determine if the areal extent of contamination has been determined, or if further delineation is necessary.

The SPLP (USEPA SPLP SW-846 Method 1312) will be used to obtain additional data to evaluate the soil to groundwater pathway and to determine appropriate screening levels for the site.

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QAPP Worksheet #18 – Sampling Locations and Methods

Sampling Location	Matrix	Depth (inches)	Analytical Group	Concentration Level	No. of Samples[1]	Sampling SOP Reference	Rationale for Sampling Location
SR859a Former Skeet Range 2	Soil	0 – 36	Metals and PAHs	Low-to-Medium	20	SOP No. 1 and No. 2	To determine the vertical and areal extent of contamination (see Worksheet #17 and Appendix D).
SR859a Former Skeet Range 2	Soil	0 – 36	SPLP for Metals and PAHs	Low-to-Medium	10	SOP No. 1 and No. 2,	
SR859a Former Skeet Range 2	Soil	0 – 36	TCLP	Low-to-Medium	5	SOP No. 1 and No. 2	Disposal Characteristics
TS862a Jeep Target Area Skeet Range	Soil	0 – 36	Metals and PAHs	Low-to-Medium	20	SOP No. 1 and No. 2	To determine the vertical and areal extent of contamination (see Worksheet #17 and Appendix D).
TS862a Jeep Target Area Skeet Range	Soil	0 – 36	SPLP for Metals and PAHs	Low-to-Medium	10	SOP No. 1, and No. 2	
TS862a Jeep Target Area Skeet Range	Soil	0 – 36	TCLP	Low-to-Medium	5	SOP No. 1 and No. 2.	Disposal Characteristics

[1] Proficiency Testing and split samples will be collected as specified by AFCEC.
TBD The number of post excavation confirmatory samples will be determined based on the boundary and volume of the excavations.

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QAPP Worksheet #19 & #30 – Sample Containers, Preservation, and Hold Times

Laboratory: Accutest Laboratories, Inc., 4405 Vineland Road, Suite C-15 Orlando, FL 32811, Jean Dent-Smith jeans@accutest.com, 407-425-6700.
List any required accreditations/certifications: DoD Environmental Laboratory Accreditation Program accreditation, compliant with the most recently published version of the DoD QSM Version 4.2.

Backup-up Laboratory: None

Sample Delivery Method: FedEx

Data Package Turnaround: 20 Days

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Sample Volume[2]	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[3] (preparation / analysis)
Soil	Metals – ICP	SW-846 6010C/ MET104/MET100	5 grams	(1)-8 oz jar	None specified per Ch.3 of SW-846	6 months
Soil	PAH	SW-846 8270D SIM/ MS008/OP007	30 grams		Cool 4°	14 days to extraction / 40 days for analysis
Soil	SPLP Metals ICP	SPLP SW-846 1312 6010C/ OP042/MET 103/MET100	30 grams	(3)-8oz jar	Cool 4°	180 days to SPLP extraction/ 180 days for analysis
Soil	SPLP PAH	SPLP SW-846 1312 / 8270D SIM OP042/OP006	30 Grams		Cool 4°	14 Days to SPLP extraction / 7 days to extraction/40 days for analysis
Soil	TCLP Metals ICP and PAHs	SW-846 1311 3010A 6010C/ OP040/MET103/MET100/MS008/OP006	30 grams		Cool 4°	180 days to TCLP extraction/ 180 days for analysis

[1] Refer to the Analytical SOP References table (Worksheet #23).
[2] The minimum sample size is based on analysis allowing for sufficient sample for reanalysis. Additional volume is needed for the laboratory MS/MSD sample analysis.
[3] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

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QAPP Worksheet #20 – Field QC Summary

Matrix	Analytical Group	No. of Field Samples[2]	No. of Field Duplicate Samples[3]	No. of Matrix Spikes[4]	No. of Matrix Spike Duplicates[4]	No. of Blanks (Trip)[5]	No. of Equipment Blanks[6]	No. of Proficiency Testing Samples	Total No. of Samples
Soil	Metals and PAH	40	4	2	2	0	10	As specified by AFCEC	48
	SPLP	10	1	1	1				13
	TCLP	5	0	0	0				5
							10		66
								Total[7]	76

[1] Specify the appropriate reference letter or No. from the Analytical SOP References table (Worksheet #23).
[2] The No. of samples collected may vary depending on field conditions.
[3] Total Numbers of Field Duplicate Samples will meet project goal of 10%.
[4] Total MS/MSD Samples will meet project goal of 5%.
[5] Trip blank samples are not required for coolers containing metals and PAH samples.
[6] Equipment blanks will be collected from non-disposable decontaminated sampling devices at a rate of 1 per day of field sampling.
[7] Post excavation confirmatory sample numbers not added to worksheet #20

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QAPP Worksheet #21 – Field SOPs

SOPs are located in Appendix A.

Reference No.[1]	Title, Revision Date and / or No.	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP No. 1	Surface and Near Surface Soil Sampling	FPM	Grab/hand auger	N	Includes descriptions and procedures for surface soil sampling.
SOP No. 2	Sub-Surface Soil Sampling	FPM	Hand Auger or Direct Push Rig	N	Includes descriptions and procedures for sub-surface soil sampling.
SOP No. 3	Sediment Sampling	FPM	Grab/hand auger	N	Includes descriptions and procedures for sediment sampling.
SOP No. 4	Surface Water Sampling	FPM	Grab	N	Includes descriptions and procedures for surface water sampling.
SOP No. 5	Sample Handling, Documentation, and Tracking	FPM	N/A	N	Includes sample packaging, shipping, and CoC requirements.
SOP No.6	Decontamination	FPM	N/A	N	Includes descriptions and procedures for decontamination of personnel and equipment.
SOP No. 7	Global Positioning System (GPS) Measurements	FPM	GPS units	N	Includes description and procedures for marking data points using GPS units.
SOP No. 8	Equipment Calibration	FPM	Various field parameter measuring equipment	N	Includes descriptions and procedures or calibrating field parameter measuring equipment.

Notes:
[1] – FPM SOPs are not project specific, as such the SOP document may include SOPs that are not relevant to the immediate project and/or tasks.

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QAPP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment	Calibration Activity	SOP Reference[1]	Responsible Person	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action
Trimble XRT2 and Nomad DGPS	No daily calibration activity. Initial receiver settings are programmed by manufacturer	SOP No. 7	Field personnel	Verify real-time location with base map on receiver and determine accuracy	Observe displayed location and actual location.	Daily	Within 0.5ft if static.	Manufacturer service

[1] The Project Sampling SOP References table is found on Worksheet #21.

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QAPP Worksheet #23 – Analytical SOP's

Laboratory SOPs are located in **Appendix B**.

SOP Reference No.	Title, Revision Date, and / or No.	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project Work? (Y/N)
MET100	Metals by ICP, Feb 2015	Definitive	Soil/Metals – ICP 6010C	Trace 6000 Series	No
MET103	Digestion of Waters for ICP Analysis, Feb 2015	Definitive	Soil Prep/Prep Method Metals – ICP SW-846 3010A	SCP Science	No
MET104	Digestion of Soils for ICP Analysis, Feb 2015	Definitive	Soil Prep/Prep Method Metals – ICP SW-846 3050B	SCP Science	No
OP006	Extraction of Semi-volatile Organics (BNAs) from Aqueous Samples, Aug 2013	Definitive	Soil Leachate/Prep Method	GlasCol shaker	No
OP007	Extraction of Semi-volatile Organics (BNAs) from Solid Samples, Aug 2013	Definitive	Soil/Prep Method	Sonic Disruptor	No
OP042	Synthetic Precipitation Leaching Of Semivolatile Organics And Metals (SPLP), Aug 2013	Definitive	Soil Leachate/SPLP Procedure Metals, Semivolatiles SW-846 1312	TCLP Tumbler	No
OP040	Toxicity Characteristic Leaching Of Semivolatile Organics And Metals (TCLP), Aug 2014	Definitive	Soil Leachate/TCLP Procedure Metals SW-846 1311	TCLP Tumbler	No
MS008	Analysis of Semivolatile Organics by method 8270D SIM, May 2014	Definitive	Semivolatiles SW-846 8270D SIM	HP6890/5973, HP6890/5975	No

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QAPP Worksheet #24 – Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Responsible Person	SOP[1]
Thermo ICAP 6000 Series	Metals, SW-846 6010C	Initial calibration daily	ICAL %RSD <5%, or Correlation coefficient R>0.995 ICV and CCV %D <10%	Instrument maintenance, nebulizer cleaning, torch inspection, standard inspection, recalibration	Laboratory Analyst	MET100
HP6890/5973 HP6890/5975	Semivolatiles, SW-846 8270D SIM, 5 points minimum	Major maintenance (per method) or second consecutive failure of opening CCV warrants recalibration	ICAL %RSD <20%, or Correlation coefficient R≥0.995; Meet minimum RF as per method.	ICAL %RSD <20%, or Correlation coefficient R≥0.995; Meet minimum RF as per method.	Laboratory Analyst	MS008

[1] The Analytical SOP References table is found on Worksheet #23.

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QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Reference SOP[1]
Trace 6000 Series	Torch, nebulizer, spray chamber, autosampler, pump tubing maintenance,	SW-846 6010C	Check connections, flush lines, clean nebulizer	Frequency determined by instrument remaining in calibration and free of interference – MET100	Passing calibration	Reconnect sample pathways, recalibrate, reanalyze affected samples	Laboratory Analyst	MET100
HP5890/5973 HP6890/5975	Injector port, column maintenance, source cleaning	SW-846 8270D SIM	Leak test, column and injector port inspection, source insulator integrity	Need for maintenance determined by passing calibration and Decafluorotriphenylphosphine (DFTPP) – see MS008	Passing DFTPP and CCV, passing Internal Standard response	Column clipping and/or reconditioning, seal and liners replacement, filaments and insulators as needed	Laboratory Analyst	MS008

[1] The Analytical SOP References table is found on Worksheet #23. Laboratory Standard Operating Procedures are subject to revision and updates during duration of the project, lab will use the most current revision of the SOP at the time of analysis.

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QAPP Worksheet #26 & #27 – Sample Handling, Custody, and Disposal

Sampling Organization: FPM Remediations, Inc.

Laboratory: Accutest Laboratories, Inc.

Method of sample delivery: FedEx

Number of days from reporting until sample disposal: 30

Activity	Organization and Title or Position of Person Responsible for the Activity	SOP Reference
Sample Labeling	FPM Remediations Inc., Field Personnel	SOP #5
Chain-of-Custody Form Completion	FPM Remediations Inc., Field Team Leader	SOP #5
Packaging	FPM Remediations Inc., Field Personnel	SOP #5
Shipping Coordination	FPM Remediations Inc., Field Personnel	SOP #5
Sample Receipt, Inspection, & Log-in	Accutest Laboratories Inc., Randy Shields	Accutest Laboratories Inc., SOPs MET100, MET103, MET104, MS008, OP042, and OP040
Sample Preparation and Determinative Analysis	Accutest Laboratories Inc., Mark Erstling (Organics), Dave Metzgar (MetalsO	Accutest Laboratories Inc., SOPs MET100, MET103, MET104, MS008, OP042, and OP040
Sample Custody and Storage	Accutest Laboratories Inc., Randy Shields	Accutest Laboratories Inc., SOPs MET100, MET103, MET104, MS008, OP042, and OP040
Sample Disposal	Accutest Laboratories Inc., Randy Shields	Accutest Laboratories Inc., SOPs MET100, MET103, MET104, MS008, OP042, and OP040

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QAPP Worksheet #28 – Analytical Quality Control and Corrective Action

Matrix	Leachate
Analytical Group	Metals
Analytical Method / SOP Reference	EPA SW-846 6010C/ MET100

QC Sample	Frequency / No.	Method / SOP QC Acceptance Limits	Corrective Action	Responsible Person	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch	<1/2 RL	The source of the contamination is investigated and eliminated before proceeding with further analysis. Corrective actions are: 1. Samples ND – report with qualification 2. Samples >10X contamination level – report with qualification 3. Samples <10x contamination – re-extract and reanalyze. Insufficient sample - qualify and footnote	Analyst/Prep analyst	Absence of interference/ contamination	<1/2 RL

QAPP Worksheet #28 – Analytical Quality Control and Corrective Action

Laboratory Control Sample (LCS)	1 per extraction batch	%Recovery = (Calculated Value/True Value) *100%; 80%<%Recovery<120%	Source of poor recovery is investigated and eliminated before proceeding with further analysis, corrective actions are: 1. Biased high, samples ND – report with qualifications. 2. Biased low – re-extract and reanalyze. Insufficient volume – qualify and footnote	Analyst/Prep analyst	Laboratory Accuracy/Method bias in ideal matrix	%Recovery = (Calculated Value/True Value) *100%; 80%<%Recovery<120%
Matrix Spike (MS)	1 per 20 samples or one for each extraction batch	%Recovery = (Calculated Value - Sample Value/True Value) *100%: 80%≤%Recovery≤120%	If the recoveries indicate that the problem is procedure related, re-extraction and re-analysis is required. If the recoveries indicate that the failures are matrix-related, refer to Blank Spike as measure of method performance in clean matrix.	Analyst/Prep analyst	Precision and Accuracy in field samples	%Recovery = (Calculated Value - Sample Value/True Value) *100%: 80%≤%Recovery≤120%

QAPP Worksheet #28 – Analytical Quality Control and Corrective Action

Matrix Spike Duplicates (MSD)	1 per 20 samples or one for each extraction batch	%Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	See above	Analyst/Prep analyst	Precision and Accuracy in field samples	%Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2
Serial Dilution Test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination.	Perform post-digestion spike (PDS) addition. Flagging criteria are not appropriate.	Analyst	Precision (field samples)	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >50x LOQ for ICP.
Post-digestion spike (PDS) addition	When dilution test fails or analyte concentration in all samples <50x MDL	Recovery within 75-125% of expected result. The spike addition should produce a level between 10x to 100x LOQ.	Run samples by method of standard additions (MSA) or apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	Analyst	Accuracy	Recovery within 75-125% of expected result. The spike addition should produce a level between 10x to 100x LOQ.

QAPP Worksheet #28 – Analytical Quality Control and Corrective Action

Matrix	Leachate
Analytical Group	PAHs
Analytical Method / SOP Reference	EPA SW-846 8270D SIM/ MS008

QC Sample	Frequency / No.	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	1 per extraction batch	<1/2 RL	The source of the contamination is investigated and eliminated before proceeding with further analysis. Corrective actions are: 1.-Samples ND – report with qualification 2.-Samples >10X contamination level – report with qualification 3.-Samples <10x contamination – re-extract and reanalyze. Insufficient sample - qualify and footnote	Analyst/Prep analyst	Absence of interference/ contamination	<1/2 RL

QAPP Worksheet #28 – Analytical Quality Control and Corrective Action

Laboratory Control Sample (LCS)	1 per extraction batch	%Recovery = (Calculated Value/True Value) *100%;	Source of poor recovery is investigated and eliminated before proceeding with further analysis, corrective actions are: 1. Biased high, samples ND – report with qualifications. 2. Biased low – re-extract and reanalyze. Insufficient volume – qualify and footnote	Analyst/Prep analyst	Laboratory Accuracy/Method bias in ideal matrix	%Recovery = (Calculated Value/True Value) *100%;
Matrix Spike (MS)	1 per 20 samples or one for each extraction batch	%Recovery = (Calculated Value - Sample Value/True Value) *100%:	If the recoveries indicate that the problem is procedure related, re-extraction and re-analysis is required. If the recoveries indicate that the failures are matrix-related, refer to Blank Spike as measure of method performance in clean matrix. The project Chemist will be contacted and a decision will be made to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed.	Analyst/Prep analyst	Precision and Accuracy in field samples	%Recovery = (Calculated Value - Sample Value/True Value) *100%:

QAPP Worksheet #28 – Analytical Quality Control and Corrective Action

Matrix Spike Duplicates (MSD)	1 per 20 samples or one for each extraction batch	%Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	See above	Analyst/Prep analyst	Precision and Accuracy in field samples	%Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2
Surrogate Spikes	Every sample	%Recovery = (Calculated Value/True Value) *100%	Reason for poor recoveries is investigated and eliminated before further analytical activities. Corrective actions are: 1.-High bias, samples ND – report with qualification. 2.-Low bias – re-extract and reanalyze. Insufficient volume – qualify and footnote	Analyst/Prep analyst	Individual sample preparation efficiency control	%Recovery = (Calculated Value/True Value) *100%

QAPP Worksheet #28 – Analytical Quality Control and Corrective Action

Internal standards (IS)	Every sample	IS Area = -50% to +100% of CCV	If failure is due to instrument performance issues, the problem must be identified, corrected, and the sample must be re-analyzed. If no instrument problem is found the sample must be re-analyzed. If upon re-analysis the responses are still not within limits, the problem may be considered sample matrix interference	Analyst	Instrument sensitivity control	Detector Stability

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QAPP Worksheet #29 – Project Documents and Records

Project Documents and Records:

Record	Generation	Verification	Storage location/archival
Field logbook and data collection sheets	FPM Field Team Leader	FPM Site Manager, Peter Corigliano	Project file and Electronic Storage, FPM office
Daily field reports			Electronic storage in project files, FPM office
Chain of Custody form	FPM Field Staff	FPM Site Manager, Peter Corigliano and Accutest Laboratories Inc., Randy Shields	Project file and Electronic storage, FPM office and Accutest permanent project records folder
Custody seals			Not stored; condition upon cooler receipt recorded on Sample Receiving Checklist
Sample labels			Not stored
Air bills	FPM Field Staff and Shipper (FedEx)	FPM Field Team Leader	Project file, FPM office
Deviations	FPM Field Team Leader and FPM Site Manager, Peter Corigliano	FPM Project Manager	Project file and Electronic Storage, FPM office
Corrective Action Reports	FPM Project Manager, Maureen Whalen	COR	Project file, FPM office
Correspondence among project team	Various	N/A	Electronic storage in project files, FPM office

Project Assessments:

Record	Generation	Verification	Storage location/archival
Field audit checklists	FPM Site Manager, Peter Corigliano	FPM Project Manager, Maureen Whalen	Project file, FPM office
Data verification checklists	FPM Chemical QC Manager, Connie van Hoesel	FPM Site Manager, Peter Corigliano and FPM Project Manager, Maureen Whalen	
Data validation report			
Data usability assessment report			

Laboratory Records

Record	Generation	Verification	Storage location/archival
Shipping Receipt or Freight Bill	Accutest Sample Receiving personnel, Randy Shields	Accutest Project Manager., Jean Dent-Smith	Initially stored in Accutest permanent project records folder; after job completion & invoicing, stored in ERPIMS
Sample Receiving Checklist			
Condition Upon Receipt Anomaly Form			
Priority form			
Chain of Custody form	FPM Field staff	Accutest Sample Receiving personnel, Randy Shields	Stored in Accutest permanent project records folder and in the Project file and Electronic Storage at FPM's office
Internal Chain of Custody Report	Accutest Sample Receiving personnel, Randy Shields	Accutest Project Manager., Jean Dent-Smith	Accutest electronic storage and ERPIMS.
Raw data files	Accutest analytical personnel, Mark Erstling (Organics), Dave Metzgar (Metals)		
Final analytical report			
Other vital records (*e.g.*, instrument maintenance records, QA records)	Accutest	Accutest	Accutest Warehouse

Laboratory Data Deliverables

Record	Metals	SVOCs
Narrative	x	x
COC	x	x
Summary Results	x	x
QC Results	x	x
Chromatograms		x

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QAPP Worksheets #31, #32, & #33 – Assessments and Corrective Action

Assessments:

Assessment Type	Responsible Party & Organization	Number/ Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Review field documentation (log book, field forms, chain-of-custody forms, etc.)	Project Manager, Maureen Whalen (FPM)	As work progresses	Will be included in RI Report	Marked-up copy of document provided to Field team leader; notify FPM Project Manager	See Worksheet #16
Field sampling audit	Project Manager, Maureen Whalen (FPM)	Once	TBD	Email or verbal report to describe the deviation from QAPP	Within 2 days of finding deficiency
Internal laboratory assessment	Accutest Project Manager, Jean Dent-Smith	Once	TBD	Documented in the laboratory report	2 weeks
External AFCEC laboratory audit PE	USAEC	Once	TBD	AFCEC report on laboratory assessment	4 weeks

QAPP Worksheets #31, #32, & #33 – Assessments and Corrective Action

Assessment Response and Corrective Action:

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Review Field Documents (Logbooks, Sampling Logs, and CoC forms)	Marked up copy of document	Maureen Whalen PM, FPM	Within 24 hours of finding deficiency	Review of corrected documentation	Field Team Leader, FPM, and Maureen Whalen PG, CPG, PMP, FPM	24 hours after notification
Field Sampling Audit	E-mail or verbal report to detail the deviation from QAPP	Maureen Whalen, PM, FPM	Within 2 days of the start of sampling	E-mail and/or phone log	Field Team Leader, FPM, and Maureen Whalen PG, CPG, PMP, FPM	2 days
Internal Laboratory Assessment	Lab Report to detail project deviations	Accutest PM	Within 5 days of sample analysis	Documented in the lab report	Accutest QA Manager	2 weeks
External AFCEC Laboratory Assessment	AFCEC findings of Laboratory project deviations	Project Chemist, FPM and Accutest Laboratory Manager	Within 7 days of analysis	AFCEC Report on Laboratory Assessment	Maureen Whalen PG, CPG, PMP, FPM, AFCEC, and Accutest Laboratory	4 weeks

QAPP Worksheet #34 – Data Verification and Validation Inputs

Item	Description	Verification (completeness)	Validation (Conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	x	
2	Contract	x	
3	Field SOPs	x	
4	Laboratory SOPs	x	
Field Records			
5	Field log books	x	x
6	Chain-of-custody forms	x	
7	Groundwater purge logs	x	x
8	Equipment calibration records	x	x
9	Relevant correspondence	x	x
10	Field audit reports	x	x
11	Field corrective action reports	x	x
Analytical Data Package			
12	Cover sheet (laboratory identifying information)	x	x
13	Case narrative	x	x
14	Internal laboratory chain-of-custody	x	x
15	Sample receipt records	x	x
16	Sample chronology	x	x
17	Communication records	x	x
18	LOD/LOQ establishment and verification	x	x
19	Standards traceability	x	x
20	Instrument calibration records	x	x
21	Definition of laboratory qualifiers	x	x
22	Results reporting forms	x	x
23	QC sample results	x	x
24	Corrective action reports	x	x
25	Raw data	x	x
26	Electronic data deliverable	x	x

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QAPP Worksheet #35 – Data Verification Procedures

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field log book	QAPP	Verify that records are present and complete for each day of field activities. Verify that all planned samples including QC samples were collected and that sample collection locations are documented. Verify that changes or exceptions were documented and reported.	Daily – Site Manager
Chain-of-custody forms	QAPP	Verify the completeness of chain-of-custody forms. Examine entries for consistency with the field logbook. Verify that the required volume of sample has been collected. Verify that sample IDs and analytes are correct and legible. Verify that all required signatures and dates are present.	Daily – Field team leader At conclusion of sampling event – Site Manager and Project Manager
Laboratory deliverable	QAPP	Verify that the laboratory deliverable contains all records specified in the QAPP. Compare the data package with the chain-of-custody forms to verify that results were provided for the correct analytes for all the collected samples. Check the sample receipt records to ensure sample condition upon receipt was noted. Review the narrative to ensure that all QC exceptions are described	Before sending to FPM – Laboratory QA Manager Upon receipt –Chemical Project QA Manager
Audit Reports, Corrective Action Reports	QAPP	Verify that all planned audits were conducted. Examine any audit reports. For any deficiencies noted, verify that corrective action was implemented according to plan.	Project QA Manager

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QAPP Worksheet #36 – Data Validation Procedures

Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator
Soil	Metals and PAHs, TCLP/ SPLP	Low-to-High	DoD QSM 4.2	Connie van Hoesel, FPM Chemical QC Manager
Soil	Metals and PAHs, TCLP/ SPLP	Low-to-High	QAPP Worksheets #12, #15 and #24. QAPP Tables 12-1 through 12-4	Connie van Hoesel, FPM Chemical QC Manager

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QAPP Worksheet #37 – Data Usability Assessment

A complete (100%) data review will be performed on the samples collected during the sampling event. The review will consist of a verification and validation based on completeness and compliance checks of sample receipt conditions and both sample-related and instrument-related QC results, as addressed in Worksheet #12. Any flags that limit the usability of the data shall be applied to all associated samples; flags are listed in Table 12-2. The Data Usability Assessment will be performed by FPM personnel. Connie van Hoesel, FPM Chemical QC Coordinator will be responsible for information in the Usability Assessment. Note that the Data Usability Assessment will be conducted on verified/validated data. After the Data Usability Assessment has been performed, data deemed appropriate for decision-making purposes will be used to assess contaminant extents at sites at Holloman AFB. The results of the Data Usability Assessment will be presented in the RI Report. The following items will be assessed and conclusions drawn based on their results.

Precision: Results of field duplicates will be presented separately in tabular format for each sample pair when results are reported above the LOD. For each field duplicate pair, the results will be assessed as stated in Table 12-3 and 12-4. MS/MSD RPDs are calculated by the laboratory and those with RPDs outside the criteria established in Table 12-1 will be listed in tabular form in the data verification report. A discussion will follow summarizing the results of the laboratory precision. Any conclusions about the precision of the analyses will be drawn and any limitations on the use of the data will be described.

Accuracy/Bias Contamination: Results for all laboratory method blanks will be evaluated and analytes detected in these blanks will be listed in tabular form in the data verification report. Laboratory data will be qualified based on the criteria listed in Table 12-3 and 12-4. A discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Overall Accuracy/Bias: Results for all LCS, surrogate and MS/MSD recoveries that are outside evaluation criteria will be presented in tabular format in the data verification reports. The results will be checked versus those listed in Table 12-1. A discussion will follow summarizing the overall accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Performance Evaluation: Performance Evaluation samples will be evaluated and if discrepancies are discovered they will be investigated and the effect on field sample results will be determined and discussed with FPM, Accutest, and AFCEC. A discussion of PE sample results will be included with QC sample discussion and aid in data defensibility. If results from PE samples are outside the expected values, an investigation will be completed to determine the source of the discrepancy. A corrective action report will be prepared to document the results of the investigation and to address whether re-sampling or reanalysis is required. If the cause is determined to also affect all samples that were collected, then an evaluation of the reliability of the field sample results will be made and reported in the data usability assessment and RI Report.

Representativeness: Representativeness is a qualitative measure of the degree to which data accurately and precisely represent a characteristic of a population, and is mainly addressed in the sample design. A measure of representativeness can also be obtained by assessing holding times

and blank data. Any conclusions about the representativeness of the samples will be drawn and any limitations on the use of the data will be described.

Comparability: In accordance with this UFP-QAPP the data are comparable when collection techniques, measurement method and reporting procedures are the same for each data set.

Completeness: A completeness check will be performed on all data generated by the laboratory. Completeness criteria are presented on Worksheet #12. Completeness will be calculated as the No. of data points for each analyte that is deemed useable (not rejected) divided by the total No. of data points for each analyte. A discussion will follow summarizing the results of the calculation of data completeness. Any conclusions about the completeness of the data will be drawn and any limitations on the use of the data will be described. Data completeness addresses only those samples that are collected and only data that is analyzed by the laboratory.

Graphics: Figures and maps will be prepared showing the site specific sampling locations and results.

Reconciliation: Each of the measurement performance criteria listed in Worksheet #12 will be examined to determine if the objective was met. Each analysis will be evaluated separately in terms of the major impacts observed from the data verification/validation, DQIs and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Usability of the data will be based on the quality assessment. After establishing the usability of the data, it will be determined if the DQO was met and if project action limits were met. The final report will include a summary of all points that comprised the reconciliation of each objective. Any conclusions or limitations on the usability of any of the data will be described.

References

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FIGURES
1 through 5

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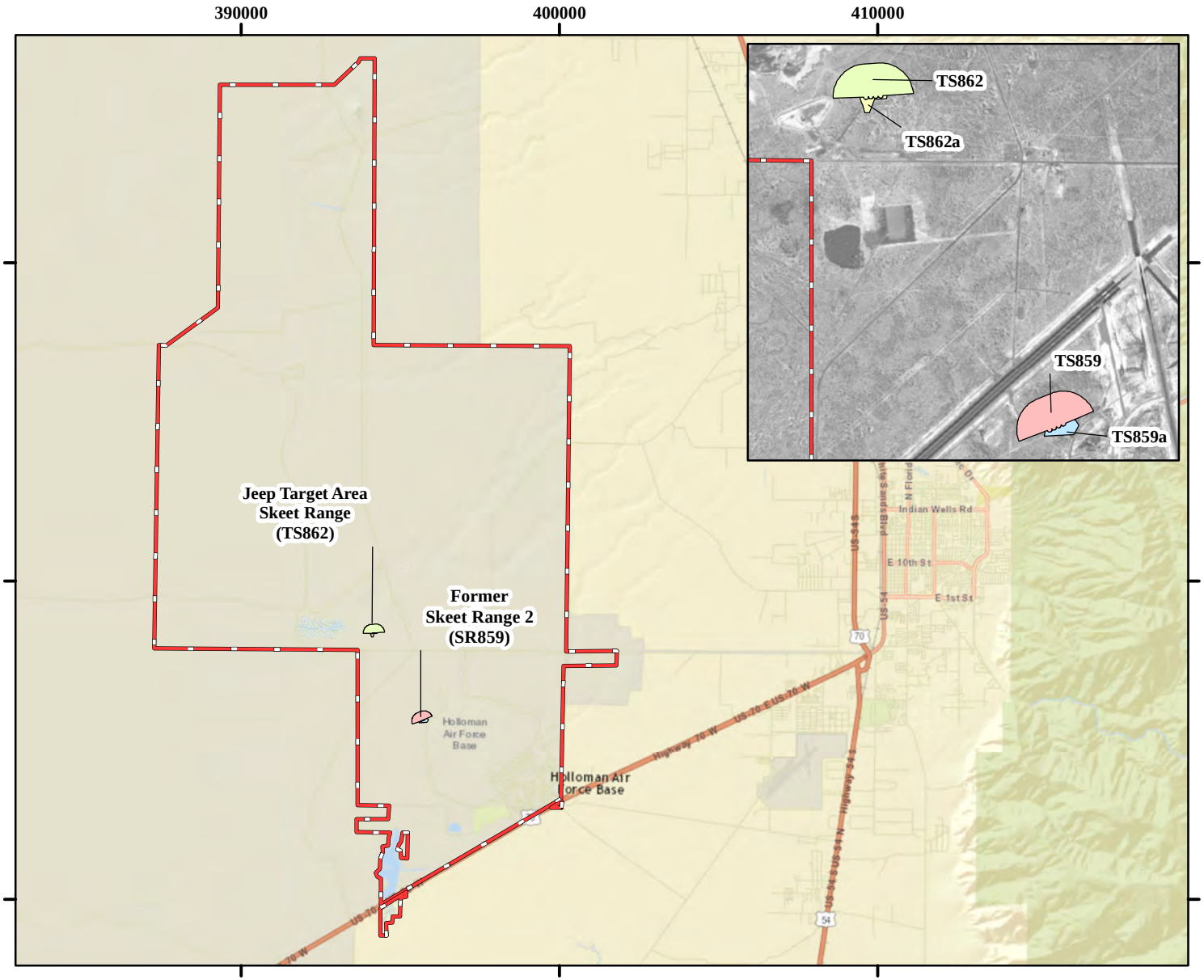
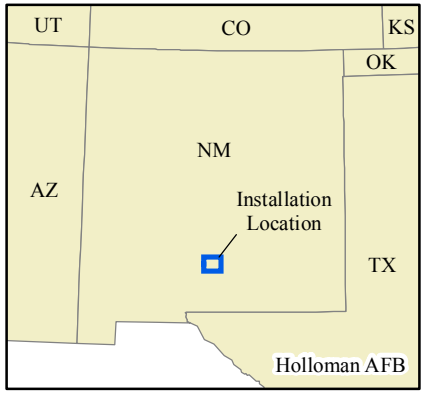




Legend

- SR859 MRS
- TS862 MRS
- SR859a MRS
- TS862a MRS
- Installation Boundary

Holloman
Air Force Base

Air Force Civil Engineer Center
2261 Huges Ave., Suite 163
Lackland AFB, Texas 78236-9853

FIGURE 1

Holloman AFB
SR859a and TS862a
Site Locations

FPM Remediations, Inc.

2015

NOTES:
Revision Date: 2/10/2015

Coordinate System:NAD 1983 UTM Zone 13N
Projection: Transverse Mercator
False Easting: 500,000.0000
Central Meridian: -105.0000
Latitude Of Origin: 0.0000
Horizontal Datum: North American 1983
False Northing: 0.0000
Scale Factor: 0.9996
Units: Meter

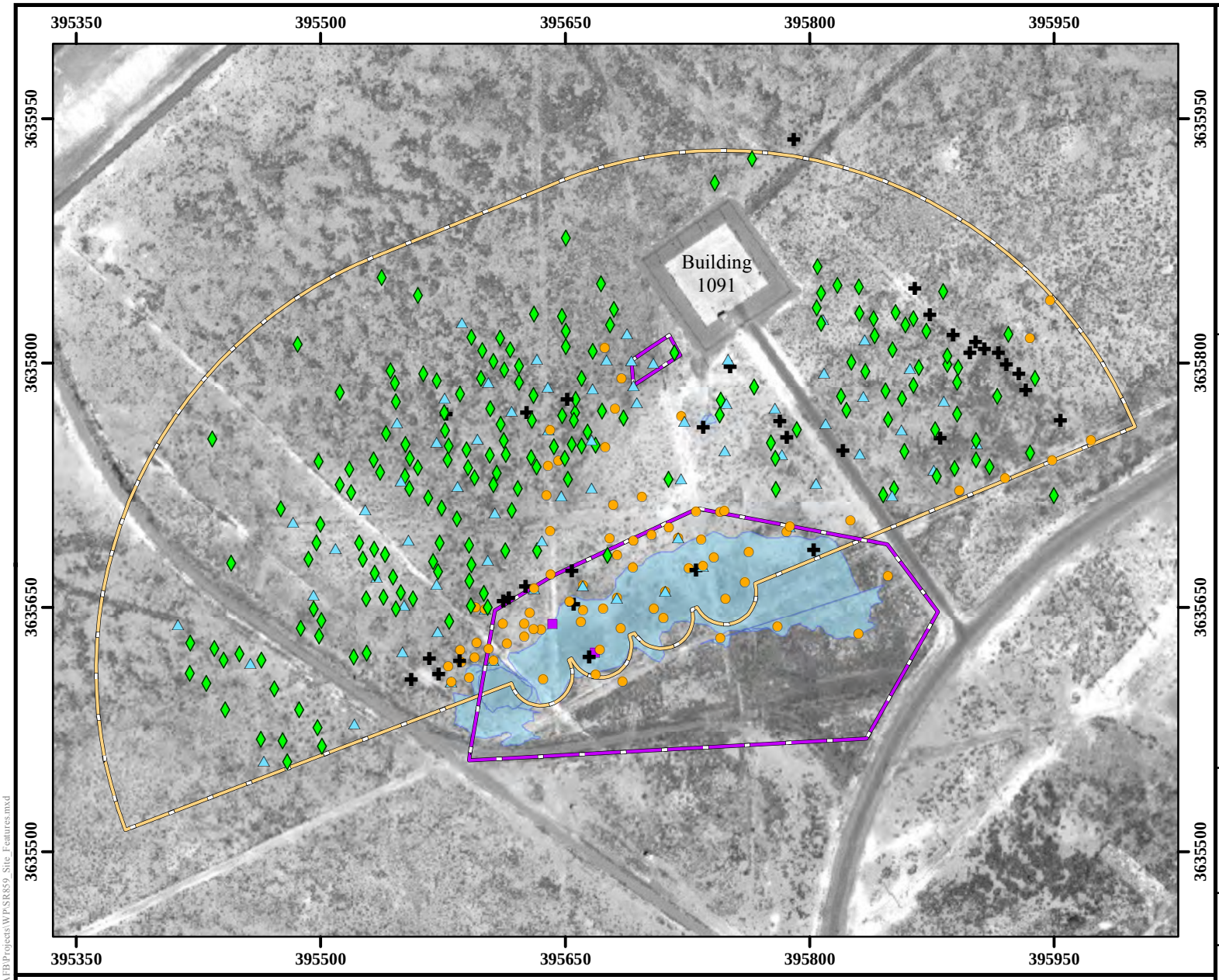
1 inch = 16,000 feet

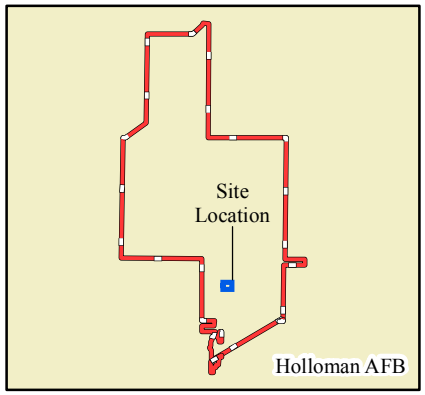
0 2.5 5 Kilometers

0 1.5 3 Miles

N W E S

Path: Y:\GIS_Projects\Holloman_AFB\Projects\WP\MRS_Locations.mxd





Legend

- Small Arms
- Clay Target Debris
- Projectile
- Lead Shot
- Blank, Casing, Shotgun Primer Cap, Shotgun Shell Wad
- SR859a MRS
- SR859 MRS
- High Density Clay Target Debris
- Installation Boundary

NOTES:

1. SR859 MRS boundary based on CSE Phase II findings.
2. Revision Date: 2/10/2015

Coordinate System:NAD 1983 UTM Zone 13N
Projection: Transverse Mercator
False Easting: 500,000.0000
Central Meridian: -105.0000
Latitude Of Origin: 0.0000
Horizontal Datum: North American 1983
False Northing: 0.0000
Scale Factor: 0.9996
Units: Meter

1 inch = 300 feet

0 45 90 Meters

0 150 300 Feet

N W E S

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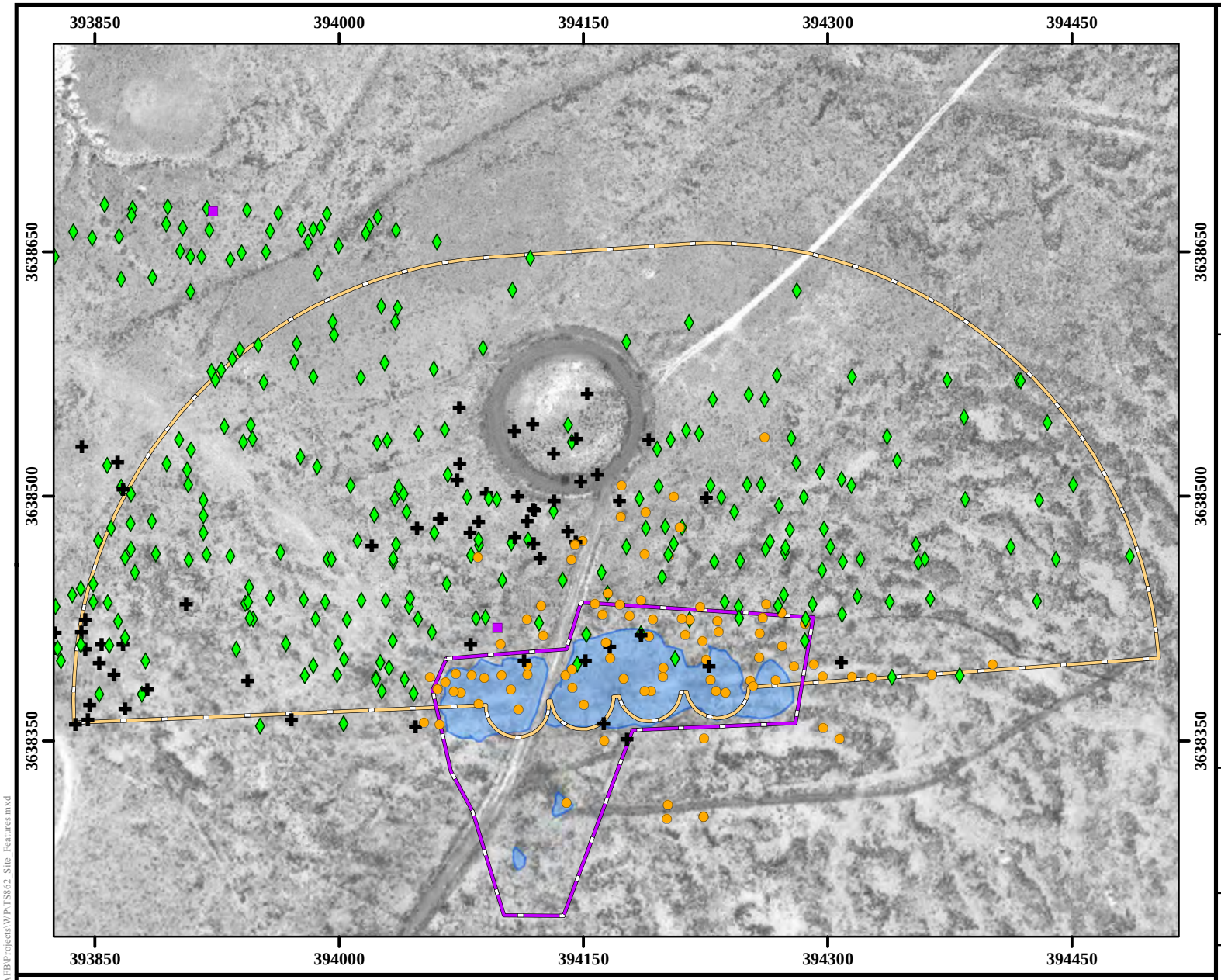
FIGURE 2

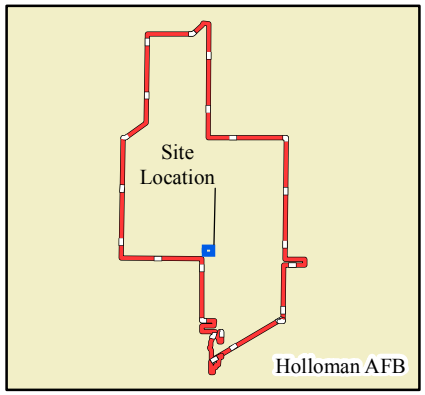
SR859a Former Skeet Range 2
Site Features

FPM Remediations, Inc.

2015

Path: Y:\GIS_Projects\Holloman_AFB\Projects\WP\SR859_Site_Features.mxd





Legend

- Small Arms
- Clay Target Debris
- Projectile
- Lead Shot
- Blank, Casing, Shotgun Primer Cap, Shotgun Shell Wad
- TS862a MRS
- TS862 MRS
- High Density Clay Target Debris
- Installation Boundary

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FIGURE 3

TS862a
Jeep Target Area Skeet Range
Site Features

FPM Remediations, Inc.

2015

NOTES:

1. SR859 MRS boundary based on CSE Phase II findings.
2. Revision Date: 2/10/2015

Coordinate System:NAD 1983 UTM Zone 13N
Projection: Transverse Mercator
False Easting: 500,000.0000
Central Meridian: -105.0000
Latitude Of Origin: 0.0000
Horizontal Datum: North American 1983
False Northing: 0.0000
Scale Factor: 0.9996
Units: Meter

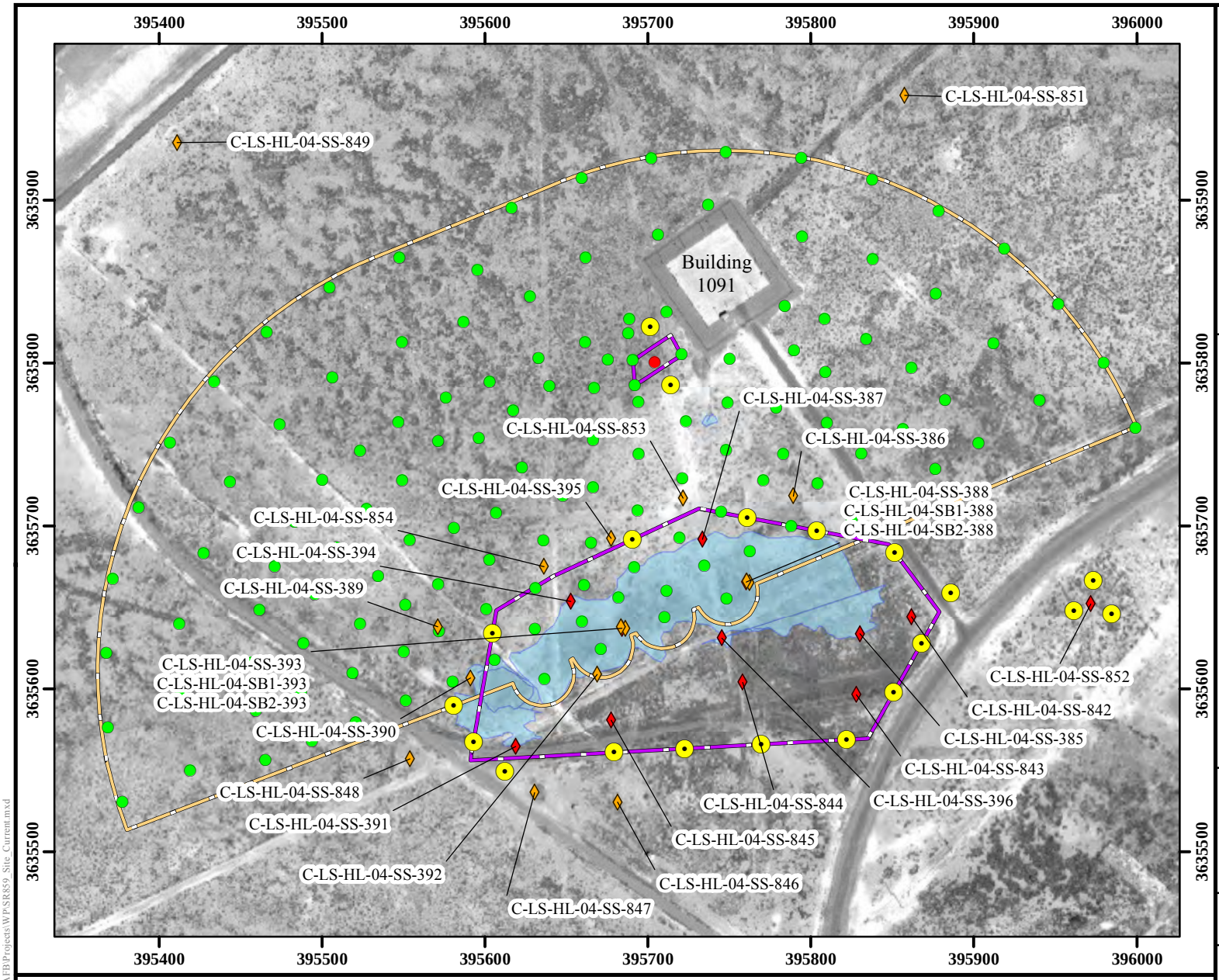
1 inch = 300 feet

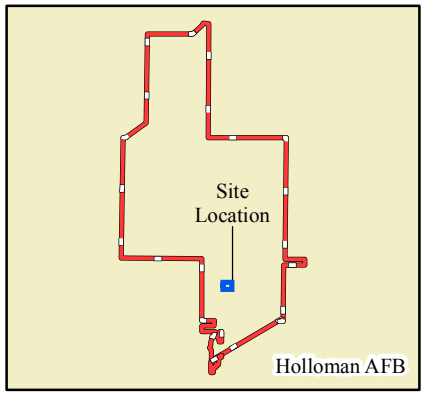
0 45.5 91 Meters

0 150 300 Feet

N W E S

Path: Y:\GIS_Projects\Holloman_AFB\Projects\WP\TS862_Site_Features.mxd





Legend

- Proposed Soil Sample Location
- PAH Sample Location Below HHSL
- PAH Sample Location Above HHSL
- Pb > 400 mg/kg
- Pb < 400 mg/kg
- SR859a MRS
- SR859 MRS
- High Density Clay Target Debris
- Installation Boundary

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FIGURE 4

SR859a Former Skeet Range 2
Proposed Sampling Locations and Historic Results

FPM Remediations, Inc.

2015

NOTES:

1. SR859 MRS boundary based on CSE Phase II findings.
2. HHSL - Human Health Screening Level.
3. Appendix E contains PAH analytical results and sample depths.
4. Revision Date: 2/10/2015

Coordinate System:NAD 1983 UTM Zone 13N
Projection: Transverse Mercator
Horizontal Datum: North American 1983
False Easting: 500,000.0000
False Northing: 0.0000
Central Meridian: -105.0000
Scale Factor: 0.9996
Latitude Of Origin: 0.0000
Units: Meter

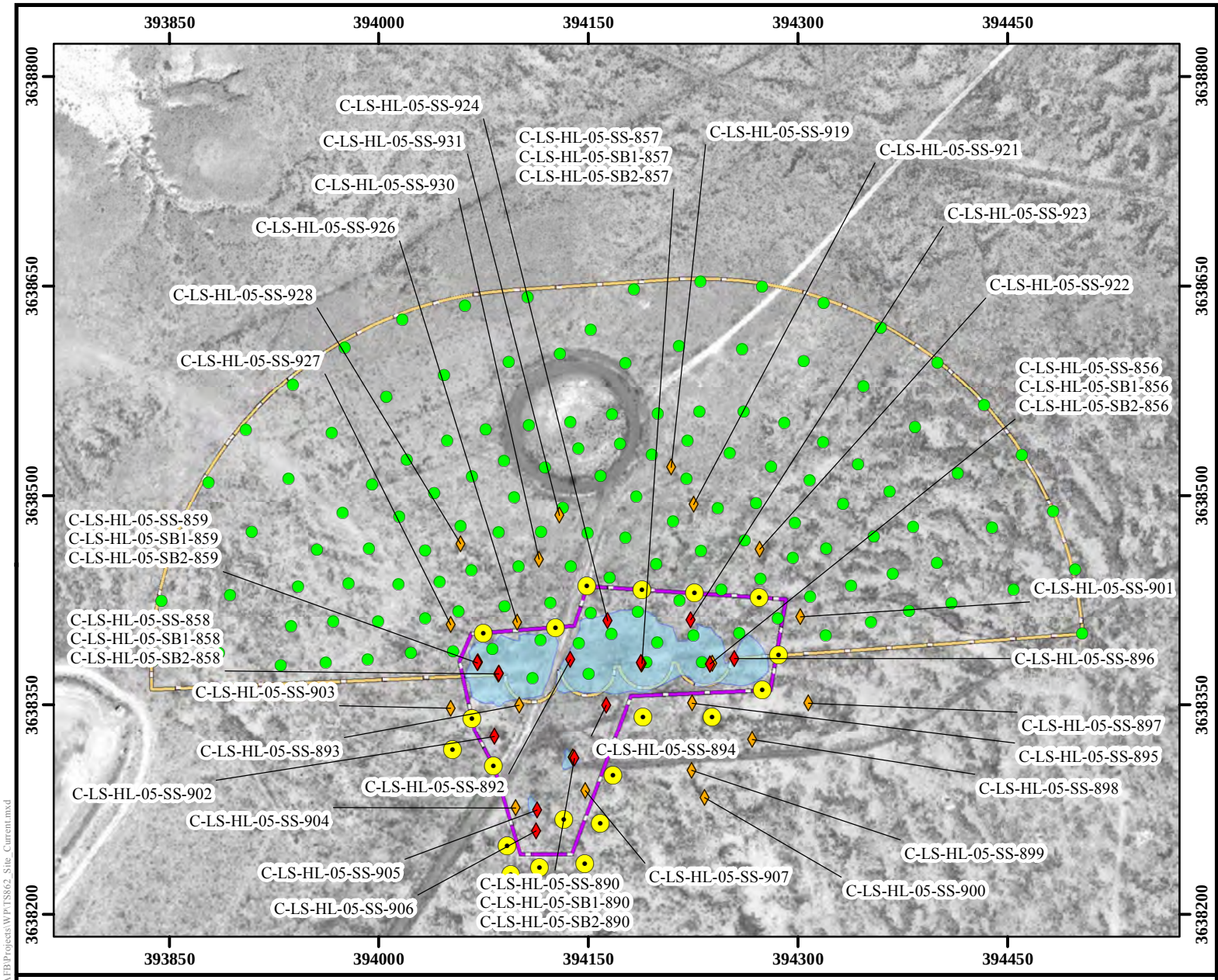
1 inch = 300 feet

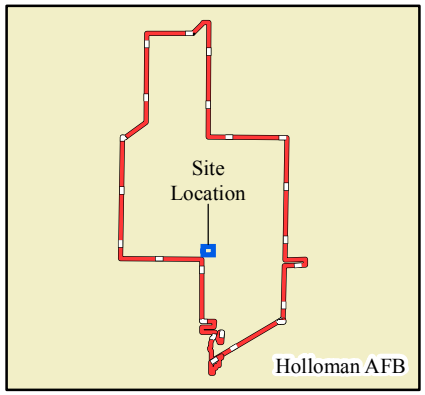
0 45 90 Meters

0 150 300 Feet

N W E S

Path: Y:\GIS_Projects\Holloman_AFB\Projects\WP\SR859_Site_Current.mxd





Legend

- Proposed Soil Sample Location
- PAH Sample Location Below HHSL
- PAH Sample Location Above HHSL
- Pb < 400 mg/kg
- High Density Clay Target Debris
- SR862a MRS
- SR862 MRS
- Installation Boundary

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FIGURE 5

TS862a Jeep Target Area Skeet Range Proposed Sampling Locations and Historic Results

FPM Remediations, Inc.

2015

NOTES:

1. SR859 MRS boundary based on CSE Phase II findings.
2. HHSL - Human Health Screening Level.
3. Appendix E contains PAH analytical results and sample depths.
3. Revision Date: 2/10/2015

Coordinate System:NAD 1983 UTM Zone 13N
Projection: Transverse Mercator
False Easting: 500,000.0000
Central Meridian: -105.0000
Latitude Of Origin: 0.0000

Horizontal Datum: North American 1983
False Northing: 0.0000
Scale Factor: 0.9996
Units: Meter

1 inch = 350 feet

0 53 106 Meters

0 175 350 Feet

N W E S

Path: Y:\GIS_Projects\Holloman_AFB\Projects\WP\TS862_Site_Current.mxd

APPENDIX A
FPM Standard Operating Procedures

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APPENDIX A
FPM Standard Operating Procedures

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1.0 SOP NO. 1 – SURFACE AND NEAR SURFACE SOIL SAMPLING

1.1 PURPOSE AND SCOPE

The purpose of this document is to define the Standard Operating Procedure (SOP) for collecting soil/sediment/groundwater/surface water samples at Holloman AFB using hand tools. This SOP describes the equipment, field procedures, and Quality Assurance/Quality Control (QA/QC) procedures implemented for sample collection.

This SOP is intended to be used together with the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) and other appropriate SOPs. Health and safety procedures and equipment for the investigation are detailed in the project Health and Safety Plan (HSP).

Applicable SOPs are listed below:

- SOP No. 5 – Sample Handling, Documentation, and Tracking
- SOP No. 6 – Decontamination
- SOP No. 7 – Global Positioning System (GPS) Measurements

1.2 EQUIPMENT AND MATERIALS LIST

The following equipment and materials should be on site for soil sampling:

- Stainless steel hand auger or hand trowel
- Surveyor's stakes and flags
- Pick
- Field logbook
- Sample Collection Field Sheets
- Nitrile gloves
- Hard plastic disposable tools (i.e., polyethylene [PE] scoop)
- Sample containers
- Sample container labels
- Label tape (clear)
- Disposable sealed zip-type PE bag
- Paper towels
- Digital camera
- 100 foot hand tape
- Waterproof and permanent marking pens
- Plastic sheeting
- Trash bags
- Cooler with sufficient ice to maintain a temperature of 4°C
- Appropriate health and safety equipment, as specified in the HSP
- Appropriate decontamination supplies, as specified in SOP No. 6

Other materials and equipment may be needed based on field conditions.

1.3 LOCATING THE SAMPLING POINTS

Sampling locations will be determined in the field. At the time of locating each sampling point, the sampling point identification will be entered in the field logbook and the GPS coordinates recorded. Information concerning nearby landmarks, or other information that will help to re-locate the point in the future will be recorded. The sample locations will be marked using surveyor's stakes and flags (or lath), and the flags (or lath) will be labeled using indelible ink with the sample point identification. A field map will be prepared as the sampling points are laid out to identify locations and tie the locations into site landmarks if available (such as foundations). If the surveyor's stake is offset from the sample location, the offset will be noted on the field map or field logbook.

1.4 SURFACE AND NEAR SURFACE SOIL DISCRETE SAMPLING PROCEDURES

Discrete samples consist of soil collected for chemical analysis from a single location. Sampling sites will be located and marked using surveying stakes or flags. Discrete surface soil and subsurface soil samples will be collected as follows:

- At each location, clear an area approximately 12 inches in diameter of surface vegetation and debris from the vicinity where a sample is to be collected.
- Use a decontaminated stainless steel spoon or disposable spoon to collect the surface soil to a depth interval of 0 to 2 inches. A steel pick may be used as needed to loosen the soil prior to sampling.
- Use a decontaminated hand auger or direct push technology to collect the shallow soil from a depth of 2 inches to 3 feet below ground surface (bgs). When proper sample depth is reached, remove the cuttings from the borehole while keeping the core intact.
- To the extent possible, eliminate gravel size or larger particles or debris based on visual observation.
- Immediately fill the appropriate sample containers. Label and handle the containers as specified in SOP No. 5, Sample Handling, Documentation, and Tracking.
- Decontaminate the sampling equipment in accordance with SOP No. 6, Decontamination.
- Once the sample is collected, the location will be documented and photographed; and GPS coordinates will be recorded.

1.5 INCREMENT SAMPLING PROCEDURES

The goal of Increment Sampling (IS) is to obtain an unbiased and reproducible estimate of the average concentration of analytes through the collection of soil sample increments distributed evenly throughout the decision unit/sampling area. Ideally, the target weight of an IS sample is approximately 1 kilogram (kg) and is comprised of 30 (minimum) to 100 increments within the decision unit. IS samples will be collected as follows:

- Determine the appropriate size of the decision unit to fit the investigation objective. Decision unit size recommendations range from 33-ft x 33-ft to 165-ft x 165-ft and consist of 30 increments to 100 increments, respectively. The location and size of individual decision units are to be based on previous investigations, visual evidence of

Munitions and explosives of concern (MEC) or Munitions Debris (MD), and/or type of MEC present.

- Using survey flags, delineate a decision unit boundary at each corner of the selected area. Note that the size and shape of the decision unit will be largely determined by the terrain features and the data quality objectives set forth in the UFP-QAPP.
- Once the boundary of the decision unit is defined, place nine flags at evenly spaced intervals along two opposite sides of the decision unit to define 10 lanes. Flags can then be used to fill in the remaining sides to create a visual sub-grid pattern. Additional flags can be placed within the interior of the decision unit if visual obstructions impede the visualization of evenly spaced increments throughout.
- With 100 increments established throughout the grid area, IS locations can then be selected. For 50-increment samples, every other flag will act as a sampling location. For 33-increment samples, every third flag will act as a sampling location. This pattern can be adjusted to satisfy the desired quantity of increments, as needed.
- Working in a team of two, one person will collect each increment while the other holds the sample container (clean plastic bag) and keeps track of the number of increments collected. The increments are sampled in a snake-like pattern from one corner of the decision unit to the corner adjacent to the starting corner.
- For the collection of QA/QC samples, the replicate samples should be collected from a sub-grid collection point offset from the original starting position and followed in the same snake-like pattern walked during the collection of the primary sample.
- Recommended sampling depths range from 1 inch to 4 inches at each increment location and are based on the overall depth distribution of anticipated analytes. The diameter of the sampling tool and the volume collected at each increment location will need to be adjusted to satisfy the 1 kg sample mass as it pertains to the selected quantity of increments in each decision unit.
- Once collected, the sample will be containerized as per the analytical laboratories requirements and labeled as specified in SOP No. 5, Sample Handling, Documentation, and Tracking.
- Once the sample collection is completed, the location will be documented and photographed; and GPS coordinates will be recorded at each of the four corners of the decision unit.

1.6 FIELD QUALITY ASSURANCE/QUALITY CONTROL SAMPLES

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

1.6.1 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. The initial sample containers for a particular parameter or set of parameters will be filled first, and then the duplicate sample containers for the same parameter(s) will be filled, and so on until

all necessary sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. Duplicate samples will be blind to the laboratory. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

1.6.2 Matrix Spike and Matrix Spike Duplicates

Matrix spikes (MS) and matrix spike duplicates (MSD) are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the chain of custody (COC) form and on the containers. It may be necessary to increase the sample volume for samples where the MS/MSD designation is to be made. If additional volume is necessary, the additional sample container will be filled immediately after the initial sample. MS/MSD samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

1.7 SAMPLE HANDLING

Sample containers, preservatives and analysis are specified in Worksheet #19. Samples will also be labeled and handled as described in SOP No. 5, Sample Handling, Documentation, and Tracking.

1.8 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on the activities concluded and also provide a permanent record of field activities. The observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages, and on field data sheets.

1.8.1 Field Sampling Data Sheet

A field sampling data sheet will be completed at each sampling location. Items not applicable to the sampling will be labeled as not applicable (NA). The information on the data sheet includes the following:

- Sampling location (and depths)
- Date and time of sampling
- Person(s) performing sampling
- Type of sample (grab or IS/Composite)
- Color (describe), odor (describe)
- Sample description
- Sample identification number
- Analyses required
- Number of sample bottles taken for each analyses
- Preservation of samples, if any
- Record of any QC samples from site
- Any irregularities or problems which may have a bearing on sample quality.

1.8.2 Field Notes

Field notes will also be kept during sampling activities. The following information will be recorded in the bound field logbook using waterproof ink:

- Names of personnel
- Weather conditions
- Date and time of sampling
- Locations, depths, and sample station numbers
- Times that procedures and measurements are completed
- Decontamination times
- Calibration information
- Calculations, if required.

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2.0 SOP NO. 2 - SUB-SURFACE SOIL SAMPLING

2.1 PURPOSE AND SCOPE

This SOP describes the equipment, materials, field procedures, and documentation procedures for collecting sub-surface soil samples using direct push or auger methods for soil characterization and chemical analysis.

Health and safety procedures and equipment to be used during soil sampling are described in a separate site-specific HSP. These SOPs are intended to be used with the Holloman AFB UFP-QAPP and with other SOPs listed below:

- SOP No. 5, Sample Handling, Documentation, and Tracking
- SOP No. 6, Decontamination

2.2 EQUIPMENT AND MATERIALS LIST

One of the following drilling equipment:

- Direct push rig (e.g., Geoprobe® rig or similar) with appropriate drilling and sampling tools (sub-surface soil)
- Hollow Stem Auger Kit and electric drill
- Hand Auger

The following equipment and materials should be on site for sub-surface soil sampling regardless of the drilling equipment used:

- Photoionization Detector (PID) (with 10.2 eV lamp)
- Weighted tape measure and ruler with 0.01-foot increments
- Surveyor's stakes and flags
- Field logbook
- Drilling Log form
- Sample Collection Field Form
- Stainless-steel bowl and spoon
- Sample containers
- Sample container labels
- Label tape (clear)
- Ziploc® bags
- Paper towels
- Digital Camera
- Waterproof and permanent marking pens
- Plastic sheeting
- Trash bags
- Ice chest with ice
- Appropriate health and safety equipment, as specified in the HSP
- Appropriate decontamination supplies, as specified in SOP No. 6

- Granular bentonite and potable water

2.3 LOCATING THE SAMPLING POINTS

The facilities designated for sampling are shown on figures provided in the UFP-QAPP. The approximate soil sampling locations will be identified on site figures before field work commences. The exact soil sampling locations will be determined in the field. Sampling coordinates will be mapped on the front of the Drilling Log in the Location Sketch/Comments Area. The sampling locations will be defined in the investigation specific work plan (WP) similar to previous investigation and long term monitoring locations.

When each soil sampling location is identified in the field, the sampling point identification will be entered in the field logbook and on the Drilling Log. Include any information concerning nearby landmarks, or other information that will help to re-locate the point in the future. Mark the sample locations using surveyor's stakes and flags, and label the flag using indelible ink with the sample point identification. A field map will be prepared as the sampling points are laid out to identify locations and tie the locations to site landmarks (such as foundations) if available. If the surveyor's stake is offset from the sample location, the offset will be noted on the field map and the field logbook.

2.4 SOIL SAMPLING PROCEDURES

At several sampling sites, the sampling locations may be in concrete or asphalt covered areas. Therefore, at these locations, cores will be drilled through the concrete or asphalt at areas most likely to contain contamination (significant cracks or low points). Direct push technology will be utilized after the concrete has been cored. Direct push samples will be collected using a dual tube sampling system or a discrete interval, piston-type sampler (Geoprobe®, MacroCore®, or equivalent). With a dual tube system, the outer rods remain in the ground while the inner rod and sample liner are extracted to retrieve a soil sample from the desired interval. Soil samples may be collected continuously throughout the depth of the direct push boring or from discrete intervals. The direct push rods will be decontaminated between boring locations, but not between samples at the same boring since a new acetate liner is used for each sample.

With a piston-type sampler, a four-foot or five-foot-long stainless steel sampler with an acetate liner is advanced to the top of the desired sampling interval. The sampler is closed to soil during advancement of the sampler to the desired sampling interval. When the top of the desired sampling interval is reached, a piston rod inside the sampler is unlocked through the drill rods, and the sampler is advanced to the bottom of the sampling interval. The sampler and all drill rods are then removed from the ground, and the acetate liner is removed from the piston sampler. Aside from the cutting shoe, the soil sampler never comes in contact with the soil sample. The cutting shoe is decontaminated after each sample is collected, and a new acetate liner is used for every sample interval. The outer sampling barrel is decontaminated after each boring is completed. The sampling will be documented in the field logbook and drill log.

With a hand auger or hollow stem auger kit, the auger head will be advanced manually to the depth. Auger extensions will be used when sampling at depths exceeding 4 feet. Once the desired depth is achieved, the auger is removed for sample collection as described below.

Following collection, the hand auger or hollow stem auger kit will be decontaminated. When using manual samplers, the sampling will be documented in the field logbook and Soil/Sediment sampling form.

At each sampling location, the sampler will be advanced by a combination of hydraulic vertical pressure and percussion hammering. Once the target depth is achieved, the sample will be withdrawn and the liner filled with the soil sample is retrieved.

The following procedures will be followed once the soil sample has been retrieved:

- Don a clean pair of nitrile gloves.
- Cut acetate sleeve to provide access to the soil sample (direct push sampling only).
- Measure the recovery. Record the sampling interval and recovery on the drilling log.
- Remove soil smear from the outside of the acetate sleeve and examine the sample, with particular attention for visible evidence of staining, odors, or other evidence of contamination. Record the soil description on the Drilling Log or Soil/Sediment Sampling Form.
- Conduct PID screening of the soil. The soil with the highest PID levels will be collected for a sample.
- The soil from the sampling interval will be removed from the liner and homogenized in a stainless-steel bowl. Once the soil has been homogenized, fill the appropriate sample containers as specified in the UFP - QAPP (Worksheet #19). Record the sample interval and analysis requested on the Drilling Log or Soil/Sediment Sampling Form and the COC.
- Label, store, transport, and document the samples (depending on the use of the sample) according to SOP No. 5. The parameters for analysis and preservation are specified in UFP QAPP Worksheet #19. All analyltical data obtained from each environmental soil sample boring shall be submitted within 30 days of receipt of laboratory results unless another schedule has been approved.
- If no other samples will be collected from the boring, abandon the boring within five days of completion by backfilling the hole with hydrated granular bentonite. Pour the granular bentonite down the hole in approximate 1-foot to 2-foot lifts, and then pour approximately 0.5 gallon of potable water down the hole to hydrate the bentonite. Continue this from the bottom of the hole to the surface. Borings greater than five feet in depth shall be completely filled from the bottom of the borehole to the ground surface with bentonite, neat cement, or 20% high solids sodium bentonite grout. The boring shall be abandoned by forced injection of grout or pouring through a tremie pipe starting at the bottom of the borehole and proceeding to the surface in one continuous operation.
- A Water Well Record Form 1903 or other form provided and/or approved document shall be completed and submitted within 30 days after abandonment.

2.5 FIELD QUALITY ASSURANCE/QUALITY CONTROL SAMPLES

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

2.5.1 Field Blanks

Field blanks are QC samples collected to evaluate potential external contamination of samples and will consist of trip, ambient, and equipment blanks. The sample collection coordinator or the project QA/QC coordinator will designate these blanks. The blanks will be assigned a QA/QC identification number, stored in an iced cooler, and shipped to the laboratory with the other samples.

A trip blank serves as a check on sample contamination originating from the container or sample transport. A trip blank consists of a VOA vial which was filled with VOA-free water at the lab, transported to the site, kept in the same cooler as the normal samples throughout the entire sampling day, and shipped back to the laboratory with the normal samples. One trip blank will be sent with each cooler containing water samples for volatile organic analyses.

The ambient blank serves as a check on sample contamination originating from ambient air during volatile organic compounds (VOCs) sample collection. An ambient blank consists of an empty VOA vial which is filled in the field with VOA free water. While pouring the sample, the water is given ample contact with ambient air conditions. The ambient blank is typically collected at the sampling location that potentially exhibits the largest ambient influence (near a busy road, airfield, etc.).

The equipment blank serves as a check on sample contamination originating from sampling equipment reuse during sample collection. The equipment blank consists of a set of sample bottles identical to the normal sample, which is filled with lab-grade water that is flushed over a decontaminated, reusable piece of equipment.

2.5.2 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. All sampling containers will be filled in the following order: volatile or gaseous analyses first, then semi-volatile organic compounds (SVOCs), including polynuclear aromatic hydrocarbons (PAHs); metals; mercury; cyanide; total organic carbon; anions; other remaining analytes (no specific order). The initial sample containers will be filled first, and then the duplicate sample containers for the same parameter(s) and so on until all sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

2.5.3 Matrix Spikes and Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) analyses are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the containers. It may be necessary to increase the sample volume for MS/MSD samples. If additional volume is necessary, the additional sample containers will be filled in the identical fashion as described above in the duplicate sample section. MS/MSD samples will be handled,

preserved, stored, and shipped in the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

2.6 FIELD DOCUMENTATION

Field documentation for sub-surface soil sampling includes field logbooks and field forms. The most important aspect of field documentation is thorough, organized, and accurate record keeping. Two forms are used in the field during sub-surface soil sampling. These forms include the Drill Log and the Soil/Sediment Sampling Form. Each form is described in Section 1.6.2. An important factor of record keeping is the proper preservation and storage of all field documentation. To preserve the field documentation, the field notes and field forms are scanned and the electronic record of the field notes is stored in the project folder and backed up on additional hard drives to prevent data loss.

Additional forms including Health and Safety Meeting forms, Health and Safety Inspection forms, and COCs used during the sampling event are detailed in SOP No. 5.

2.6.1 Field Logbook

All information pertinent to soil sampling and not documented on the field forms will be recorded in a bound field logbook with consecutively numbered pages. The field logbook notes will be recorded in indelible ink. The field logbooks notes are entered to create an accurate record of the work performed so that the sampling activity can be reconstructed without relying on the memory of field personnel. Information documented in the field logbook may include information on date of notes, weather conditions, field personnel, site, mobilization, work performed including location and time, etc. After each day, field notes are reviewed by the field team leader or site responsible person for accuracy. Refer to SOP No. 5 for detailed procedures regarding documentation in the field logbook.

2.6.2 Field Forms

<u>Surface Water Sampling Form</u>

- Field personnel
- Project name and number
- Site Identifier
- Sample Location Identifier
- Sizes and types of sampling equipment
- Date of sample
- Water parameters
- A description of the color and odor.
- Comments or Observations
- Sample Identifier
- Sample Collection Time

Soil/Sediment Sampling Form

The Soil/Sediment Sampling Form contains the following minimum information:

- Field personnel
- Project name and number
- Site Identifier
- Sample Location Identifier
- Sizes and types of sampling equipment
- Date of sample
- Sampling depth.
- A description of the recovered soil sample. The descriptions should include origin, grain size, texture, structure, color, and odor.
- Comments or Observations
- Sample Identifier
- Sample Collection Time

3.0 SOP NO. 3 – SEDIMENT SAMPLING

3.1 PURPOSE AND SCOPE

This SOP describes composite and point sampling procedures to be used in the collection of surface and near surface sediment samples.

This SOP is intended to be used together with the UFP-QAPP and other appropriate SOPs. Health and safety procedures and equipment for the investigation are detailed in the project HSP. Applicable SOPs are listed below:

- SOP No. 5, Sample Handling, Documentation, and Tracking
- SOP No. 6, Decontamination
- SOP No. 7, GPS Measurements

3.2 EQUIPMENT AND MATERIALS LIST

General equipment used for collecting sediment samples includes:

- Laboratory provided sample containers and labels
- Bound field logbook
- Sample data sheets
- Plastic flagging
- Stainless steel hand auger and/or hand trowel
- Surveyor's stakes and flags
- Shovel (if sampling rocky areas)
- Nitrile gloves
- Stainless steel mixing bowl and spoon
- Waterproof, permanent marking pens
- Plastic sheeting
- Trash bags
- Label tape (clear)
- Ziploc® bags
- Paper towels
- Digital camera
- Cooler with sufficient ice to maintain a temperature of 4°C
- Appropriate health and safety equipment, as specified in the HSP
- Appropriate decontamination supplies, as specified in SOP No. 6

3.3 LOCATING THE SAMPLING POINTS

Sampling locations will be determined in the field. At the time of locating each sampling point, the sampling point identification will be entered in the field logbook and the GPS coordinates recorded. Information concerning nearby landmarks, or other information that will help to re-locate the point in the future will be recorded. The sample locations will be marked using surveyor's stakes and flags (or lath), and the flags (or lath) will be labeled using indelible ink with the sample point identification. A field map will be prepared as the sampling points are laid

out to identify locations and tie the locations into site landmarks if available (such as foundations). If the surveyor's stake is offset from the sample location, the offset will be noted on the field map or field logbook.

3.4 SEDIMENT SAMPLING PROCEDURES

Section 3.4.1 describes the procedures for sample collection, compositing of samples, and for collecting grab samples.

3.4.1 Grab and Composite Sediment Sampling

Grab sediment samples will be collected at sampling locations determined by the results of the visual survey and geophysical program. The grab samples will, in most cases, be co-located with surface water sample locations. When this occurs, the grab sediment sample location should also be suitable for surface water sampling, if possible. Once identified, the grab sample point should be marked for sample collection and surveying.

The grab sediment sample will be collected from the interval between the mud line surface and 6 inches below the surface following these procedures:

- Decontaminate all sediment sampling equipment prior to sample collection and between samples in accordance with SOP No. 6, Decontamination.
- Collect representative sediments from the 0 to 6 inch interval below the mud line using a stainless steel coring tool or trowel and transfer into the stainless steel mixing bowl. Grab samples will consist of sediment collected from a single location. For composite sample collection, sediment will be collected from five or more "grab" locations (ie. "spoke and hub" method), and added to the stainless steel mixing bowl. The remaining procedure will be followed identically for both grab and composite sampling.
- If sediments are to be sampled at locations where surface water samples are to be collected, the surface water sample should be collected first to avoid disturbance of the mud line.
- Remove any cobbles, large pebbles, vegetation, or other material from the sediment.
- Scrape the sediment from the sides, corners, and bottom of the stainless steel mixing bowl with a stainless steel spoon or disposable spoon and roll the sediment to the center of the bowl.
- Thoroughly mix the sediment sample until the mixture is as homogeneous as possible. Note: if water is present in the sample mixture that is obtained by use of a stainless steel spoon or disposable spoon, an attempt will be made to preserve the water-to-solid ratio by including the water as part of the sample.
- Use the stainless steel spoon or disposable spoon to place the homogeneous sample/water mixture into each sample container.
- Remix the sample/water mixture remaining in the mixing bowl after each scoop of material is placed in a sample container. This approach is used to evenly distribute the liquid into the sample container, while attempting to maintain the solid-to-liquid ratio present in the sample collected.

- If the mixture is high in liquid content, pour the mixture into a stainless steel beaker to more efficiently transfer the mixture into the sample containers. Continue mixing with a spoon to maintain homogeneity of the sample mixture during filling of sample containers.
- Decontaminate the outside surface of all sample containers.
- Place the samples in sealable freezer bags.
- Place the samples on ice in the cooler.
- Complete field documentation and chain-of-custody form(s).
- Decontaminate sampling equipment in accordance with SOP No. 6, Decontamination.

3.5 FIELD QUALITY ASSURANCE/QUALITY CONTROL SAMPLES

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

3.5.1 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. The initial sample containers for a particular parameter or set of parameters will be filled first, and then the duplicate sample containers for the same parameter(s) will be filled, and so on until all necessary sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. Duplicate samples will be blind to the laboratory. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

3.5.2 Matrix Spike and Matrix Spike Duplicates

MS and MSDs are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the containers. It may be necessary to increase the sample volume for samples where the MS/MSD designation is to be made. If additional volume is necessary, the additional sample container will be filled immediately after the initial sample. MS/MSD samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

3.6 SAMPLE HANDLING

Sample containers, preservatives and analysis are specified in Worksheet #19. Samples will also be labeled and handled as described in SOP No. 5, Sample Handling, Documentation, and Tracking.

3.7 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on the activities concluded and also provide a permanent record of field activities. The observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages, and on field data sheets.

3.7.1 Field Sampling Data Sheet

A field sampling data sheet will be completed at each sampling location. Items not applicable to the sampling will be labeled as not applicable (NA). The information on the data sheet includes the following:

- Sampling location (and depths)
- Date and time of sampling
- Person(s) performing sampling
- Type of sample (grab or composite)
- Color (describe), odor (describe)
  Sample description
- Sample identification number
- Analyses required
- Number of sample bottles taken for each analyses
- Preservation of samples
- Record of any QC samples from site
- Any irregularities or problems which may have a bearing on sample quality.

3.7.2 Field Notes

Field notes will also be kept during sampling activities. The following information will be recorded in the bound field logbook using waterproof ink:

- Names of personnel
- Weather conditions
- Date and time of sampling
- Locations, depths, and sample station numbers
- Times that procedures and measurements are completed
- Decontamination times
- Calibration information
- Calculations, if required.

4.0 SOP NO. 4 – SURFACE WATER SAMPLING

4.1 PURPOSE AND SCOPE

This SOP describes procedures to be used in the collection of water samples from water bodies, ponds, and seeps. This procedure sets forth the methods for collection of samples at any site based upon physical characteristics and dimensions of the water body.

This SOP addresses the collection of representative ambient water quality samples that meet applicable regulations and appropriate sampling protocols and is intended to be used with the UFP-QAPP, and with other SOPs listed below:

- SOP No. 5, Sample Handling, Documentation, and Tracking
- SOP No. 6, Decontamination

4.2 EQUIPMENT AND MATERIALS LIST

Sample containers will be obtained from the analytical laboratory. Several extra bottles will be obtained in the event of breakage, use for temporary storage of samples to be filtered, or for solving other sampling issues.

Equipment that may be used during surface water sample collection includes:

- Long-handled PE, or glass sample cup
- PE, or glass surface water grab sampler
- Multi-parameter water quality meter (Horiba U-52 or YSI 556 or equivalent). Parameters shall include: pH/temp, electrical conductivity, DO, oxidation reduction potential, and turbidity
- PE or glass jar for field measurement samples
- Surveyor's stakes and flags
- Nitrile gloves
- Measuring tape with weighted end (for lake/pond sampling only)
- Field logbook
- Field Sampling Forms
- Sample containers (with preservatives if required)
- Sample container labels
- Plastic squeeze bottle filled with deionized or distilled water
- Label tape (clear)
- Ziploc® bags
- Paper towels
- Digital camera
- Waterproof, permanent marking pens
- Trash bags
- Cooler with sufficient ice to maintain a temperature of 4°C
- Appropriate health and safety equipment, as specified in the HSP

- Appropriate decontamination supplies, as specified in SOP No. 6

4.3 LOCATING THE SAMPLING POINTS

Sampling locations will be determined in the field. At the time of locating each sampling point, the sampling point identification will be entered in the field logbook and the GPS coordinates recorded. Information concerning nearby landmarks, or other information that will help to re-locate the point in the future will be recorded. The sample locations will be marked using surveyor's stakes and flags (or lath), and the flags (or lath) will be labeled using indelible ink with the sample point identification. A field map will be prepared as the sampling points are laid out to identify locations and tie the locations into site landmarks if available (such as foundations). If the surveyor's stake is offset from the sample location, the offset will be noted on the field map or field logbook.

4.4 PROCEDURES FOR SURFACE WATER AND STREAM SAMPLING

4.4.1 General Sampling Procedures

The following general procedures will be followed to collect surface water samples:

- Decontaminate sampling equipment according to SOP No. 6
- Don a clean pair of nitrile gloves.
- Collect water quality measurements using a multi-parameter water quality system prior to sampling. Measurements will include pH, temperature, specific conductivity, DO, ORP, and turbidity. Measurements will be recorded on the field sampling form immediately.
- Collect surface water sample utilizing methods described in Sections 6.4.2 and 6.4.3.
- Individual sample bottles should be filled in the order given below:

    1. VOC
    2. Alkalinity
    3. SVOC (includes explosives)
    4. Metals
    5. Mercury
    6. Cyanide
    7. Total Organic Carbon
    8. Anions
    9. Other remaining analytes

- VOC sample vials should be completely filled so the water forms a convex meniscus at the top, then capped so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, discard the sample vial and repeat the procedure until no air bubbles appear.
- Alkalinity sample bottles are also collected so that no air space remains in the vial. Turn the vial over and tap it to check for bubbles in the vial, which indicate air space. If air bubbles are observed in the sample vial, add additional water until no air bubbles appear.
- Fill bottles for SVOCs, metals and other analytes until almost full.

- Record time of sample collection on the associated field form.

4.4.2 Pond Sampling Procedures

A primary objective of sampling a pond or other lentic body of water is to collect a water sample that is representative of current water quality conditions. Additionally, when sampling in a pond, the sample should be representative of the sampled depth and location. To collect a sample from depth, a water-sampling bottle that can be triggered from the surface is essential.

The equipment listed in Section 6.2 should be available for use when sampling from a pond. When selecting a sampling bottle, always consider the following: 1) what analytes and levels of detection are included in the analysis program, and 2) what total volume of water is needed for all anticipated analysis. Use a non-metallic sampling bottle to avoid contamination of metals analyses.

Samples will be collected by the "container immersion" method or the "dip and transfer" method. Water samples will be collected by filling the collection container or transfer device held just beneath the surface of the water, unless deeper samples are required. If a transfer device is used, the sample will be poured directly from the beaker or dipper into the sample collection container. If volatile analysis is to be performed, deliver water to the volatiles container very slowly to minimize potential volatile loss. If field meters used to measure field parameters do not have cords long enough to reach the sampling depth, deliver water into a clean beaker and measure the required field parameters. Fill out the appropriate field sampling forms being sure to include the sampling depth.

Label the laboratory sample bottles appropriately, decontaminate all sample bottle exteriors, and immediately transfer to the cooler containing ice. Move to the next sampling location and repeat aforementioned sampling procedures.

4.4.3 Stream and Seep Sampling Procedures

The field sampling team will obtain samples from steams and seeps by placing the sample containers near the center of flow taking care to minimize solid matter being transferred into the containers. The "dip and transfer" method or "container immersion" method will be utilized when sufficient sample volume is present. However, if the flow conditions are slow and volume is limited, a PE or glass bowl or pan can be placed near the center of flow until sufficient volume has accumulated for analysis (for slow seep conditions).

If slow flow conditions are present and adequate sample volume cannot be collected immediately using a sample container but can be obtained within 2 hours, the following procedure may be used:

1. Dig a small depression in the soil within the flow path, if necessary.
2. Place the rim of a PE or glass bowl or pan below the discharge point.
3. Allow the bowl or pan to fill.
4. Transfer the water into an appropriate laboratory container.

5. Repeat steps 2 through 5 until adequate sample volume is obtained (includes field parameter measurements).

Alternatively, the following procedure may be used for low flow seeps and should be used for circumstances in which adequate sample volume cannot be collected within 2 hours:

1. A funnel or similar sample collection device is placed within the flow path.
2. The stem of the funnel is positioned such that flow of water passes directly into a relatively closed sample collections device such as a sample bottle. The funnel stem may also be connected to tubing which in turn transfers the water into a relatively closed sample collection device such as a sample bottle.
3. If the sample collection device is a sample bottle containing a preservative, do not allow the sample bottle to overflow.

Field parameters will be measured on an aliquot of sample and recorded for each sample location.

If flow conditions are so low that a sample cannot be collected, the sampler should record on the field sampling form that seep flow conditions prevented sample collection at that location.

Label the laboratory sample bottles appropriately, decontaminate all sample bottle exteriors, and immediately transfer to the cooler containing ice. Move to the next sampling location and repeat aforementioned sampling procedures.

4.5 FIELD QUALITY ASSURANCE/QUALITY CONTROL SAMPLES

Field QA/QC samples are designed to help identify potential sources of external sample contamination and evaluate potential error introduced by sample collection and handling. All QA/QC samples will be labeled with QA/QC identification numbers and sent to the laboratory with the other samples for analyses.

4.5.1 Duplicate Samples

Duplicate samples are samples collected to assess precision of sampling and analysis. Duplicate samples will be collected at the same time and for the same parameters as the initial samples. The initial sample containers for a particular parameter or set of parameters will be filled first, and then the duplicate sample containers for the same parameter(s) will be filled, and so on until all necessary sample containers for both the initial sample and the duplicate sample have been filled. The duplicate samples will be handled, preserved, stored, and shipped in the same manner as the primary samples. Duplicate samples will be blind to the laboratory. The rate of duplicate sample collection is specified in the UFP-QAPP (Worksheet #20).

4.5.2 Matrix Spike and Matrix Spike Duplicates

MS and MSDs are used to assess the potential for matrix effects. Samples will be designated for MS/MSD analysis on the COC form and on the containers. It may be necessary to increase the sample volume for samples where the MS/MSD designation is to be made. If additional volume is necessary, the additional sample container will be filled immediately after the initial sample. MS/MSD samples will be handled, preserved, stored, and shipped in the same manner as the

primary samples. The rate of MS/MSD collection is specified in the UFP-QAPP (Worksheet #20).

4.6 SAMPLE HANDLING

Sample containers, preservatives and analysis are specified Worksheet #19. Samples will also be labeled and handled as described in SOP No. 5, Sample Handling, Documentation, and Tracking.

4.7 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on the activities concluded and also provide a permanent record of field activities. The observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages, and on field data sheets.

4.7.1 Field Sampling Data Sheet

A field sampling data sheet will be completed at each sampling location. Items not applicable to the sampling will be labeled as not applicable (NA). The information on the data sheet includes the following:

- Sampling location (and depths)
- Date and time of sampling
- Person(s) performing sampling
- Type of sample (grab or composite)
- Water quality parameters
- Color (describe), odor (describe)
- Sample description
- Sample identification number
- Analyses required
- Number of sample bottles taken for each analyses
- Preservation of samples
- Record of any QC samples from site
- Any irregularities or problems which may have a bearing on sample quality.

4.7.2 Field Notes

Field notes will also be kept during sampling activities. The following information will be recorded in the bound field logbook using waterproof ink:

- Names of personnel
- Weather conditions
- Date and time of sampling
- Locations, depths, and sample station numbers
- Times that procedures and measurements are completed
- Decontamination times
- Calibration information
- Calculations, if required

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5.0 SOP NO. 5 – SAMPLE HANDLING, DOCUMENTATION, AND TRACKING

5.1 PURPOSE AND SCOPE

This SOP describes the procedures for sample handling, documentation, and tracking. This SOP is intended to be used with the UFP-QAPP, and with other SOPs listed below:

- SOP No. 1 – Surface and Near Surface Soil Sampling
- SOP No. 2 – Sub-Surface Soil Sampling
- SOP No. 3 – Sediment Sampling
- SOP No. 4 – Surface Water Sampling

5.2 SAMPLE IDENTIFICATION

The sampling locations, sample types, and naming conventions will be established prior to field activities for each sample to be collected. On-site personnel will obtain assistance in defining any special sampling requirements from the FPM Project Manager or designated Task Manager. Each sample will have a discrete, alpha-numeric sample identification (ID). A unique sample ID is needed to track each of the samples collected for analysis during the life of this project. In addition, the sample IDs will be used in the database to identify and retrieve the analytical results received from the laboratory. Each sample ID will be assigned at the time of sampling.

Sample ID

The sample ID will be designated as follows: Site Code, Sample Type and Sampling Location Indicator, Sample Location Number, Sample Depth Identifier, and Sample Type Qualifier.

Site Code

The first segment consists of two to five alphanumeric characters that designate the site code. Examples of site codes include:

- SR for Former Skeet Range 2
- TS for Jeep Target Area Skeet Range

For the surface sample designated “SRSS0101AA”, The “SR” indicates that the site from which the sample is collected. For a soil sample designated “TSSO0101AA”, “TS” indicates the sample is collected from site Jeep Target Area Skeet Range.

Sample Type and Sampling Location Indicator

The second segment consists of one or two alphanumeric characters that indicate the sample type and sampling location indicator. Sample types are as shown below:

SO – Surface Soil (0 to 2 inches)
SS – Subsurface Soil (2 inches or greater)
IS – Increment Soil Sample
SD – Sediment
M – Groundwater from monitoring well sampling locations
T – Groundwater from direct-push groundwater samples that were not completed as permanent monitoring wells (i.e., temporary well point
SW – Surface Water

For the surface soil sample designated "SRSO0101AA", The "SO" indicates that the sample is collected from a surface soil location. For a soil sample designated "TSSS0101AA", "SS" indicates the sample is collected from subsurface soil.

Sample Location Number

The two-digit number following the sample indicator completes the identification of the sampling location at a specific site.

For the sample designated "SRSO0101AA", The "01" indicates that the sample is collected from location 1.

Sample Depth Identifier

The fourth segment consists of two numerical characters that will be used to identify the depth in feet below top of inner casing in wells and feet bgs for soil samples.

For a soil sample designated "TSSO0101AA", "01" indicates the sample is collected 1 foot bgs.

Sample Type Qualifier

The fifth segment is two alphabetic characters used to designate the type of sample. The first letter denotes the round of sampling completed (e.g., "A" for first quarterly sampling round, "B" for second quarterly sampling round, etc.). The sample types will be identified by the second character as listed below:

- A = Primary sample
- B = Primary sample
- C = Field duplicate groundwater sample
- D = Matrix Spike Duplicate (MSD)
- E = Equipment blank
- F Ambient blank
- R = Trip blank
- S Matrix Spike (MS)

The letter A or B appearing at the end of a sample number indicates that the sample is a primary sample. These letters will be selected randomly to mask the predominance of primary samples

over QA/QC samples. This system was devised to minimize the likelihood that the laboratory personnel can distinguish the primary samples from the QA/QC samples using the sample identification.

5.3 SAMPLE LABELS

Sample labels will be filled out as completely as possible by a designated member of the sampling team prior to beginning field sampling activities each day. All sample labels will be filled out using waterproof ink. At a minimum, each label will contain the following information:

- Sampler's company affiliation
- Site location
- Sample ID
- Date and time of sample collection
- Analyses required
- Method of preservation (if any) used
- Sample matrix (i.e., soil, surface water)
- Sampler's signature or initials

5.4 SAMPLE HANDLING PROCEDURES

This section discusses proper sample containers, preservatives, and handling and shipping procedures. The UFP-QAPP summarizes the information contained in this section and also includes the sample holding times for each analyte.

5.4.1 Sample Containers

Certified, commercially clean sample containers will be obtained from the contract analytical lab. The contract laboratory will label the containers to indicate the type of sample to be collected. Required preservatives will be prepared and placed in the containers at the laboratory prior to shipment to the site. Appropriate sample containers for the specific analyses required will be listed in the UFP-QAPP.

5.4.2 Sample Preservation

Sample preservation efforts will commence at the time of sample collection and will continue until analyses are performed. Samples will be stored on ice at 4°C in coolers immediately following collection. The ice will be double bagged in plastic storage bags. Additional sample preservation requirements are listed in the UFP-QAPP. Chemical preservatives, if necessary, will be added to the sample containers by the laboratory prior to shipment to the field, unless otherwise specified in the UFP-QAPP.

5.4.3 Sample Handling and Shipping

The sample containers will be wiped clean of all sample residue and then wrapped in protective packing material (bubble wrap) and taped. Samples will be double-bagged with plastic bags and then placed upright in an iced cooler. Additional packing material will be placed around the samples as necessary to protect them from damage and to keep them upright. A COC form will accompany each cooler. The COC will be placed in a plastic bag and attached to the inside lid of the cooler. The cooler lid will be taped closed with a custody seal.

Coolers will be hand delivered or shipped by overnight express carrier to the analytical laboratory. All samples must be shipped for laboratory receipt and analyses within specific holding times. This may require daily shipment of samples with short holding times. The condition of all samples as received and temperature of all coolers will be reported by the laboratory.

5.4.4 Holding Times and Analyses

The holding time is specified as the maximum allowable time between sample collection and analysis and/or extraction, based on the analyte of interest and stability factors, and preservative (if any) used. Allowable holding times are listed in the UFP-QAPP.

5.5 SAMPLE DOCUMENTATION AND TRACKING

This section describes documentation required in the field notes, on the sample collection field sheets, on the daily quality control reports, and on the sample COC forms.

5.5.1 Field Logbook

All entries in logbooks will be made in waterproof ink and corrections will consist of line-out deletions that are initialed and dated. Field investigation situations vary widely. No general rules can include each type of information that must be entered in a logbook for a particular site. A site-specific logging procedure will be developed to include sufficient information so that the sampling activity can be reconstructed without relying on the memory of field personnel. The logbooks will be kept in the field team member's possession or in a secure place during the investigation. Following the investigation, the logbooks will become a part of the final project file.

The following information (as applicable) shall be recorded in the field log book:

- Sampler’s printed name and signature
- Names of other field personnel (FPM and any FPM subcontractors) and site visitors
- Date (month, day, year)
- General weather conditions

- Time and location of sampling (including approximate distance to adjacent landmarks if possible)
- Level of personal protective equipment (PPE) used
- Brief description of sampling method with references to appropriate SOPs and site-specific WP
- Sample ID (includes location and matrix)
- Any QA/QC sample
- Number and volume of sample containers and requested analysis
- Sample handling and preservation
- Results of any field measurements, equipment used, and equipment calibration information
- Decontamination information
- Brief discussion of any field decisions, unusual conditions, problems encountered and corrective action taken, and/or changes required by field conditions
- Signature and date by person responsible for writing the field notes

5.5.2 Daily Quality Control Report

Each sampling crew will also maintain DQCRs to supplement the information recorded in the field logbook. DQCRs will be maintained by members of the field sampling team and cross-checked for completeness at the end of each day by the sampling team members and/or Field Manager. They will be signed and dated by individuals making entries and initials by the reviewer upon completion. Copies of the DQCR will be forwarded to the Quality Assurance Officer for review. The DQCR will include the following information:

- Project name
- Project number
- Personnel on site
- Visitor on site
- Subcontractors on site
- Equipment on site
- Weather conditions
- Field work performed
- Quality control and health and safety activities
- Problem, down time, and standby time
- Name and title of person completing the DQCR

5.5.3 Sample Chain of Custody

During field sampling activities, traceability of the sample must be maintained from the time that the samples are collected until laboratory data are issued. Initial information concerning collection of the samples will be recorded in the field logbook as described above. Information on the custody, transfer, handling, and shipping of samples will be recorded on a COC form. The COC form used in the field is a one-page form.

The sampler will be responsible for initiating and filling out the COC form. The sampler will sign the COC when the sampler relinquishes the samples to anyone else. One COC form will be completed for each cooler of samples collected daily. The COC will contain the following information:

- Sampler's signature and affiliation
- Project number
- Date and time of collection
- Sample identification number
- Sample type
- Analyses requested
- Number of containers
- Signature of persons relinquishing custody, dates, and times
- Signature of persons accepting custody, dates, and times
- Method of shipment
- Shipping air bill number (if appropriate)

The person responsible for delivery of the samples to the laboratory will sign the COC form, and retain a copy of the COC form, document the method of shipment, and send the original and the second copy of the COC form with the samples. Upon receipt at the laboratory, the person receiving the samples will sign the COC form and return the second copy to the FPM Chemical Quality Control Coordinator. Copies of the COC forms documenting custody changes and all custody documentation will be received and kept in the central files. The original COC forms will remain with the samples until final disposition of the samples by the laboratory. The analytical laboratory will dispose of the samples in an appropriate manner 60 to 90 days after data reporting. After sample disposal, a copy of the original COC will be sent by the laboratory to the FPM Chemical Quality Control Coordinator to be incorporated into the central files.

6.0 SOP NO. 6 – DECONTAMINATION

6.1 PURPOSE AND SCOPE

This SOP describes the equipment, materials, field procedures, and documentation procedures for decontaminating sampling equipment and personnel. Health and safety procedures and equipment to be used during soil sampling are described in a separate HSP. The procedures presented below are intended to be used with other SOPs listed below:

- SOP No. 1 – Surface and Near Surface Soil Sampling
- SOP No. 2 – Subsurface Soil Sampling
- SOP No. 3 – Sediment Sampling
- SOP No. 4 – Surface Water Sampling

The overall objective of an environmental sampling program is to obtain samples that accurately depict the chemical, physical, and/or biological conditions at the sampling site. Extraneous contaminants can be brought onto the sampling location and/or introduced into the medium of interest during the sampling program (e.g. using sampling equipment that is not properly or fully decontaminated). Trace quantities of contaminants can consequently be captured in a sample and lead to false positive analytical results and, ultimately, to an incorrect assessment of the contaminant conditions associated with the site. Decontamination of sampling equipment (e.g., all non-disposable equipment that will come in direct contact with samples) and field support equipment (e.g., drill rigs, vehicles) is, therefore, required prior to, between, and after uses to ensure that sampling cross-contamination is prevented, and that on-site contaminants are not carried off-site.

6.2 EQUIPMENT AND MATERIALS LIST

The following is a list of equipment that may be needed to perform decontamination:

- Brushes
- Wash tubs
- Buckets
- Scrapers, flat bladed
- Hot water – high-pressure sprayer
- Sponges or paper towels
- Alconox detergent (or equivalent)
- Potable tap water or distilled water
- Laboratory-grade de-ionized water
- Garden-type water sprayers
- Appropriate Health and Safety equipment (i.e., nitrile gloves, safety glasses, etc.)
- Appropriate containers for Investigative Derived Waste (IDW)

6.3 DECONTAMINATION PROCEDURES

Site activities should be conducted with the general goal of preventing the contamination of personnel and equipment. FPM sampling personnel contaminated and, therefore, reduce the need and extent of decontamination. However, some type of decontamination will always be required on site. A sample personnel decontamination will use remote sampling techniques, bag monitoring instruments, avoid contact with obvious contamination, and employ dust suppression methods as necessary to reduce the probability of becoming set-up guideline and a sample decontamination equipment and supplies list are included in the HSP.

6.3.1 Decontamination Solutions

A decontamination solution should be capable of removing, or converting to a harmless substance, the contaminant of concern without harming the object being decontaminated. The preferred solution is a mixture of detergent and water, which is a relatively safe option compared to chemical decontaminants. A solution recommended for decontaminating consists of 1 to 1.5 tablespoons of Alconox per gallon of warm water. Skin should be decontaminated by washing with hand soap and water. The decontamination solution must be changed when it no longer foams or when it becomes extremely dirty. Rinse water must be changed when it becomes discolored, begins to foam, or when the decontamination solution cannot be removed.

6.3.2 Personnel Decontamination

A temporary personnel decontamination line will be set up in the Contamination Reduction Zone, which is outside of the Exclusion Zone where intrusive work is being performed. If contamination is not encountered, a dry decontamination station may be established which consists of an area where disposable PPE can be donned and discarded. It is anticipated that all work at Holloman AFB will be completed in Level D.

6.3.3 Sampling Equipment Decontamination

The following steps will be used to decontaminate sampling equipment:

- Personnel will dress in suitable safety equipment to reduce personal exposure as required by the HSP.
- Gross contamination on equipment will be scraped off at the sampling or construction site.
- Equipment that cannot be damaged by water will be placed in a wash tub containing Alconox or low-sudsing non-phosphate detergent along with potable water and scrubbed with a bristle brush or similar utensil. Equipment will be rinsed with tap water in a second wash tub followed by a de-ionized or distilled water rinse.
- Equipment that may be damaged by water will be carefully wiped clean using a sponge and detergent water and rinsed with de-ionized or distilled water. Care will be taken to prevent equipment damage.

Following decontamination, equipment will be placed in a clean area or on clean plastic sheeting to prevent contact with contaminated soil. If the equipment is not used immediately after decontamination, the equipment will be covered or wrapped in plastic sheeting, foil, or heavy-duty trash bags to minimize potential contact with contaminants.

6.3.4 Equipment Leaving the Site

Vehicles used for activities in non-contaminated areas shall be cleaned on an as-needed basis, as determined by the UXO Site Safety Officer/QC Supervisor (UXOSO/QCS), using soap and water on the outside and vacuuming the inside. On-site cleaning will be required for very dirty vehicles leaving the area.

6.3.5 Responsible Authority

Decontamination operations at each hazardous waste site shall be supervised by the UXOSO/QCS. The UXOSO/QCS is responsible for ensuring that all personnel follow decontamination procedures and that all contaminated equipment is adequately decontaminated. The UXOSO/QCS is also responsible for maintaining the decontamination zone and managing the wastes generated from the decontamination process.

6.3.6 Investigation Derived Waste

Liquid wastewater from decontamination will be left on the temporary decontamination pads and allowed to evaporate. Solid waste, including sample liners and PPE, will be removed from the site and properly disposed of.

6.4 EMERGENCY DECONTAMINATION

Emergency decontamination procedures should be followed if necessary to prevent the loss of life or severe injury. In the case of threat to life, decontamination should be delayed until the victim is stabilized; however, decontamination should always be performed first, when practical, if it can be done without interfering with essential lifesaving techniques or first aid, or if a worker has been contaminated with an extremely toxic or corrosive material that could cause severe injury or loss of life. During an emergency, provisions must also be made for protecting medical personnel and disposing of contaminated clothing or equipment.

6.5 DOCUMENTATION

Sampling personnel will be responsible for documenting the decontamination of sampling and drilling equipment. The documentation will be recorded with waterproof ink in the sampler's field notebook with consecutively numbered pages. The information entered in the field book concerning decontamination will include the following:

- Decontamination personnel
- Date and start and end times
- Decontamination observations
- Weather conditions
- IDW handling

7.0 SOP NO. 7 - GLOBAL POSITIONING SYSTEM MEASUREMENTS

7.1 PURPOSE AND SCOPE

This SOP provides technical guidance and methods that will be used to perform GPS measurements at the field site.

GPS surveying at the field site is used to record:

- Locations of MEC or MD
- Excavation footprints
- Sampling locations
- Injection locations
- Other surface and subsurface feature locations and elevations

The procedures presented below are intended to be used with other SOPs listed below:

- SOP No. 1 – Surface and Near Surface Soil Sampling
- SOP No. 2 – Sub-Surface Sampling
- SOP No. 3 – Sediment Sampling
- SOP No. 4 – Surface Water Sampling

7.2 PERSONNEL QUALIFICATIONS

GPS measurements at the field site will be performed by qualified field personnel. All personnel engaged in recording GPS measurements will be knowledgeable and experienced in methods and equipment use.

7.3 GPS SURVEYING

GPS equipment capable of achieving measurement precision of equal to or less than the specified accuracy without correction will be used. GPS equipment should collect data such that post-processing of spatial data can be performed to increase measurement precision, if needed. The equipment will be operated in accordance with manufacturer's specification, operations manual, and generally accepted surveying practices.

Surveying equipment will be field-verified each day before beginning surveying by establishing the coordinates of a known location (ie, temporary benchmark) using the GPS unit. The benchmark identification (or description) and measured coordinates will be recorded in the survey logbook.

7.3.1 Survey Points

GPS equipment will be used to record the grid corner and center-point coordinates, that will be marked for future reference during the investigation. GPS will be used to record other pertinent site feature data, for example the location of MEC or MD and anthropogenic material, if encountered. Prior to collecting the center-point sampling location coordinates, each location will be marked with a survey flag. The sample location ID will be recorded on each survey flag. Sample locations will be measured from the center of the grid cell or grab location. For each GPS location recorded an identifier and the coordinates will be stored in the data logger.

If the coordinates at a survey location cannot be determined due to the presence of tree cover or other obstacles which prohibit adequate signal reception, coordinates will be obtained at a minimum of two alternate locations (offsets) close to the original survey location. The distance and bearing from each of the alternate locations to the original survey location will then be determined using a measuring tape and compass.

7.3.2 Coordinate Systems

It is assumed all GPS measurements will be recorded in the UTM coordinate system in a zone consistent with the longitudinal boundaries of the given site location. The horizontal datum will be North American Datum of 1983 (NAD83). The vertical datum will be North American Vertical Datum of 1988 (NAVD88). The site-specific grid will be referenced to known National Geodetic Survey (NGS) benchmarks, if possible.

7.3.3 Required Accuracy

At a minimum, surveyed location coordinates will be determined to an accuracy of ±0.5 foot. Vertical elevations measured by GPS are suspect due to limited system accuracy. Accuracy will be assessed using the FGDC Geospatial Positioning Accuracy Standards. Data may be post-processed to increase accuracy, if required.

7.4 DOCUMENTATION

The field team is responsible for documenting all survey measurements. A complete and accurate record correlating the sample IDs to the instrument assigned stations IDs will be kept in the field logbook. The observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages, and on field data sheets as applicable. Upon completion of each day's fieldwork, the electronic record will be downloaded from the instrument, correlated with the sample IDs, and uploaded into the project database.

8.0 SOP NO. 8 - EQUIPMENT CALIBRATION

8.1 PURPOSE AND SCOPE

This SOP describes the procedures for equipment calibration and documentation. This SOP is intended to be used with the UFP-QAPP, FSP and with other SOPs listed below:

- SOP No. 1 – Surface and Near Surface Soil Sampling
- SOP No. 2 – Sub-Surface Soil Sampling
- SOP No. 3 – Sediment Sampling
- SOP No. 4 – Surface Water Sampling

8.2 EQUIPMENT AND MATERIALS LIST

The following section provide a list of equipment that may be needed to perform equipment calibration.

Horiba U-22 and Horiba U-52:

- Horiba U-22
- Horiba U-52
- Auto calibration solution pH 4
- Calibration cup
- Calibration log for Horibas

YSI 556

- YSI 556
- Calibration cup
- Calibration log for YSI
- DI water
- Conductivity solution (1.413 µS/cm)
- pH 4 solution
- pH 7 solution
- ORP solution (240 mV)
- PID, miniRAE
- PID, miniRAE
- Tedlar bag
- Isobutylene (100 ppm)
- Calibration log for PID

8.3 EQUIPMENT CALIBRATION PROCEDURES

The following provides the procedures for the calibration of the Horiba U-22 and U-52, YSI 556, and PID miniRAE.

Horiba U-22:

- Turn on Horiba.
- Place probe in auto calibration solution (pH 4.00).
- Press Cal button.
- Press Ent button, calibration begins.
- END appears when calibration is complete.
- Press MEAS button and collect pH reading.
- The acceptable pH range is 3.96 to 4.04.
- If any errors appear, refer to Horiba U-22 manual.

Horiba U-52:

- Turn on Horiba.
- Place probe in auto calibration solution (pH 4.00).
- Press Cal button.
- Press Ent button, calibration begins when the parameters on screen start to blink.
- When parameters stop blinking, calibration is complete.
- Collect pH reading.
- The acceptable pH range is 3.96 to 4.04.
- If any errors appear, refer to Horiba U-52 manual.

YSI 556:

- Turn on YSI 556.
- Press ESC which will lead to main menu.
- Scroll to Calibrate and press ENT.
- Scroll to DO, press enter, scroll to DO%
- Enter barometric pressure.
- Place probe in DI water (in calibration cup) and loosely tighten probe to calibration cup.
- Press enter, and then enter again.
- DO% is instantly calibrated.
- Acceptable range is 95% to 105%.
- Press ESC to return to calibration menu.
- Scroll to Conductivity, press enter, scroll to Conductivity in list and press enter
- Enter standard, 1.413 µs/cm.
- Fill calibration cup with conductivity solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.

- Conductivity is instantly calibrated.
- Acceptable range is 1.408 to 1.418 µs/cm.
- Press ESC to return to calibration menu.
- Scroll to pH, press enter, scroll to 2-point calibration and press enter
- Enter 1st standard, 4.00.
- Fill calibration cup with pH 4.00 solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- pH is instantly calibrated.
- Acceptable range is 3.95 to 4.05.
- Press enter.
- Enter 2nd standard, 7.00.
- Fill calibration cup with pH 7.00 solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- pH is instantly calibrated.
- Acceptable range is 6.95 to 7.05.
- Press ESC to return to calibration menu.
- Scroll to ORP, press enter
- Enter standard, 240 mV.
- Fill calibration cup with ORP solution.
- Place probe in solution and tighten probe to calibration cup.
- Press enter, and then enter again.
- Conductivity is instantly calibrated.
- Acceptable range is 235 to 245 mV.
- If any errors appear, refer to YSI 556 manual.

PID miniRAE:

Zero Calibration

- Turn on PID to Zero Calibration menu.
- Press [Y/+] to start calibration.
- Press [MODE] to quit and return to the main calibration display.
- Zero calibration starts.
- When Zero calibration is complete, you see this message: Zeroing is done!, Reading = 0.000 ppm.

Span Calibration

- Turn on PID to Scan Calibration menu.
- The span gas is first be filled into a Tedlar bag.
- Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.
- Press [Y/+] to enter Span calibration.
- Turn on your span calibration gas.

- Press [Y/+] to initiate calibration.
- Span calibration starts and displays this message: Calibrating...
- When Span calibration is complete, you see this message: Span 1 is done!, Reading = 100.0 ppm.

Per the Mini RAE manual, there is no set range of what is allowed above or below 100 ppm. The Manual simply states that the "reading should be very close to the span gas value".

8.4 DOCUMENTATION:

Documentation for equipment calibration forms which are included in Daily CQCRs. The calibration forms include:

- Equipment model and number
- Date
- Calibration personnel
- Standard calibration values
- Scan gas concentration for PID calibration
- Standard calibration solution parameters for water quality

APPENDIX B
Laboratory Certifications and Standard Operating Procedures
(CD Only)

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LABORATORY ACCREDITATION BUREAU

Certificate of Accreditation

ISO/IEC 17025:2005 Certificate Number L2229

Accutest Laboratories Southeast, Inc.

4405 Vineland Road, Suite C-15
Orlando FL 32811

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: December 15, 2015

R. Douglas Leonard, Jr., President, COO
Laboratory Accreditation Bureau
Presented the 29th of January 2013

*See the laboratory's Scope of Accreditation for details of accredited parameters
**Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

Prepared by:	Dave Metzgar	Date:	08/28/2013
Reviewed by:	Svetlana Izosimova	Date:	09/13/2013

Annual Review

Reviewed by:		Date:	
Reviewed by:		Date:	
Reviewed by:		Date:	

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Issued to:		Date:	
Issued to:		Date:	
Issued to:		Date:	

Effective 7 days after "*" date

TEST NAME: METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY

INSTRUMENT: THERMO 6500, SERIAL # 20100903 SSTRACE 1
INSTRUMENT: THERMO 6500, SERIAL # 20103825 SSTRACE 2
AUTOSAMPLER: CETAC 240 POSITION, SERIAL # 031038A520 SSTRACE 1
AUTOSAMPLER: CETAC 240 POSITION, SERIAL # 041048A520 SSTRACE 2

SUGGESTED WAVELENGTH (S): TABLE 2

METHOD REFERENCES: SW846 6010C, EPA 200.7 Rev 4.4 1994

DEPARTMENT: Metals

REVISIONS: Section 2.0: added pH is checked within metals department
Section 3.0: added detail
Section 5.8: added PCOS and instrument software information
Section 6.6.2: changed “values” to “concentrations” and added detail
Section 6.7: remove references to CRI
Section 7.12: removed reference to CRI
Section 7.13: removed entire section
Section 8.5: removed entire section
Table 6: removed CRI section

1.0 SCOPE AND APPLICATION SUMMARY

1.1 This method is applicable for the determination of metals in water, sludges, sediments, and soils. Elements that can be reported by this method include: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Titanium, Thallium, Tin, Vanadium, and Zinc.

1.2 Sample matrices are pretreated following SW846 and EPA methods for digestion of soil, sediment, sludge or water samples. Refer to specific metals department digestion SOP's for more information on digestion techniques.

1.3 This inductively coupled argon plasma optical emission spectrometer (s) (ICP-OES) uses an Echelle optical design and a Charge Injection Device (CID) solid-state detector to provide elemental analysis. Control of the spectrometer is provided by PC based iTEVA software. In the instrument, digested samples are introduced into the Thermo 6500 ICP, passed through a nebulizer and transported to a plasma torch. The element-specific emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a spectrometer, and the intensities of the emission lines are monitored with the solid state detector.

1.4 Reporting limits (RL) are based on the extraction procedure. Reporting limits may vary depending on matrix complications, volumes and by client needs, but the reporting limits must always be verified with a low check which meets the criteria outlined in this SOP. Solid matrices are reported on a dry weight basis. Refer to table 1 of this SOP for Accutest Southeast typical reporting limits. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits.

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1.5 MDLs must be established for all analytes, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs must be determined approximately once per year for each matrix and instrument. Please refer to Accutest QA SOP QA020, current version for further information regarding method performance criteria and experimental method detection limits.

1.6 An MDL check standard will be analyzed at the time of the annual MDL study and on a quarterly basis for verification. The concentration of the MDL check standard must be 1x-4x the statistical MDL. The MDL Check Standard is carried through the entire preparation and analytical procedure. This is a qualitative check; therefore, the analyte needs to be detected only. If the analyte is not detected, the concentration of the MDL check standard must be increased to a level where the analyte is detected. This then becomes the current MDL.

1.7 Lower limit of quantitation check sample. The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on a quarterly basis to demonstrate the desired detection capability. The LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within 20 percent of their true value.

1.8 MDLs are generated for each matrix on both ICP instruments. The higher of the two statistically calculated MDL's is entered into LIMS as the MDL. The verified MDLs are stored in the LIMS and must be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.9 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

1.10 Instrument Detection Limits (IDL). It is suggested that IDL's be completed upon initial instrument installation and whenever instrument conditions have significantly changed. The Instrument Detection Limits (in ug/L) are determined by analyzing 7 replicates of a reagent blank solution on 3 non consecutive days. The IDL is defined as 3 times the average of the standard deviation of the 3 days. Each IDL measurement shall be performed as though it were a separate analytical sample. IDLs shall be determined and reported for each wavelength used in the analysis of the samples.

2.0 PRESERVATION AND BOTTLEWARE

All samples should be preserved with nitric acid to a pH of <2 at the time of collection. All sample pH are checked in sample receiving and within the metals department. Samples that are received with a pH >2 must be preserved to pH <2 and held for 24 hours prior to metals digestion to dissolve any metals that absorb to the container walls. Refer to SOP SAM101, current revision for further instruction. Final pH of TCLP extracts are checked and recorded in Accutest Southeast Extractions Department. Please refer to TCLP (1311) fluid determination logbook and SPLP (1312) fluid determination logbook for further information. TCLP extracts

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received from Accutest Southeast Extractions Department are prepared as soon as possible, no longer than 24 hours from time of receipt. If precipitation is observed during the sample preparation process the sample(s) are immediately re-prepped on dilution until no precipitation is observed. Samples received for dissolved metals analysis should be filtered and preserved to pH<2 within 72 hours of collection. Refer to Accutest Southeast Sample Filtration Logbook for further information.

All soil samples must be stored in a refrigerator at $\leq 6^{o}C$ upon receipt. Refer to SOP SAM101, current revision for further instruction.

All bottleware used by Accutest Southeast is tested for cleanliness prior to shipping to clients. Analysis results must be less than one half the reporting limit to be acceptable. Refer to SOP SAM104, current revision for further instruction.

3.0 HOLDING TIME AND BATCH SIZE

All samples must be prepared and analyzed within 6 months of the date of collection. Refer to appropriate Accutest Southeast digestion SOP, current revision for batch size criteria.

4.0 INTERFERENCES

Several types of interferences can cause inaccuracies in trace metals determinations by ICP. These interferences are discussed below.

4.1 Spectral interferences are caused by overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and background contribution from stray light from the line emission of high concentration elements. Corrections for these interferences can be made by using interfering element corrections, by choosing an alternate analytical line, and/or by applying background correction points. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

Note: Refer to section 17.0 of this SOP for further instruction regarding interfering element correction factor generation.

4.2 Physical interferences can be caused by changes in sample viscosity or surface tension, by high acid content in a sample, or by high dissolved solids in a sample. These interferences can be reduced by making sample dilutions.

4.3 Matrix interferences in high solid samples can be overcome by using an internal standard. Yttrium/Indium mix is used for the Thermo 6500 ICP. The concentration must be sufficient for optimum precision but not so high as to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation.

4.4 Chemical interferences are not pronounced with ICP due to the high temperature of the plasma, however if they are present, they can be reduced by optimizing the analytical conditions (i.e. power level, torch height, etc.).

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5.0 APPARATUS

5.1 Currently there are two solid state ICPs available for use in the lab. Both are Thermo 6500 ICP units. These units have been optimized to obtain lower detection limits for a wide range of elements. Since they are solid state systems, different lines may be included for elements to obtain the best analytical results. However, the lines which are normally included in the normal analysis program are shown in Table 2.

5.2 Instrument auto samplers. For random access during sample analysis.

5.3 Class A volumetric glassware and pipettes.

5.4 Polypropylene auto sampler tubes.

5.5 Eppendorf Pipette (s) - Pipette (s) are checked for accuracy and to ensure they are in good working condition. Volumes are checked at 100% of maximum volume, the 50% (mid-range) and between 10% and 25% at the low range, whichever constitutes most frequently used volume for a particular pipette. Pipettes are checked within the metals department approximately once per week and stored electronically in the “Eppendorf Calibration Log”. Refer to SOP QA006, current revision for further information regarding pipette calibration.

5.6 Fisher Brand 0.45 micron (um) filter or equivalent. Filter lots are checked for cleanliness through the Method Blank process. All Method Blank analytical results must be less than one half the reporting limit to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated filters must be re-filtered through acceptable filters.

5.7 Fisher Brand disposable 10 ml syringes or equivalent. Syringe lots are checked for cleanliness through the Method Blank process. All Method Blank results must be less than one half the reporting limit to be acceptable, if not , the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated syringes must be re-filtered through acceptable syringes.

5.8 Data System

Microsoft Windows XP Professional Version 2002
Instrument software – Thermo iTEVA version 2.5.0.84

5.8.1 A computer system interfaced to the Thermo 6500 ICP that allows for the continuous acquisition and storage of all data obtained throughout the duration of the analytical run sequence.

5.8.2 Data is archived to a backup server for long term storage.

6.0 REAGENTS

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All chemicals listed below are trace metal grade unless otherwise specified. Refer to Acid Certificate of Analysis logbook for Certificates of Analysis and compliance with the specifications of the grade listed. Accutest Laboratories produces DI water to the specifications for the ASTM Type II standard designation based on the system manufacturer's performance specifications. The DI water is used exclusively for laboratory purposes. De-ionized (DI) water should be used whenever water is required. Refer to SOP QA037, current revision for more information regarding testing and monitoring. Refer to the Metals Department Standard Prep Logbook for the make-up and concentrations of standards and stock solutions being used within this SOP. Some of the information included in the logbook is as follows: standard name, elements in mix, manufacturer, lot number, parent expiration date, acid matrix, stock concentration, volume of standard added, total volume, final prepared concentration, prep date, initials, MET number, and prepared standard expiration date. Standards and prepared reagents must be prepared every 6 months or before stock standard expiration date, whichever comes first**.** Refer to tables 3 through 7 of this SOP for concentration levels of standards used. Unless otherwise approved, the calibration curve must contain 3 points determined by a blank and a series of standards representing the elements of interest.

6.1 2.5 ppm Yttrium and 10 ppm Indium internal standard, made from ICP quality standard.

6.2 Hydrochloric acid, trace metals grade.

6.3 Nitric Acid, trace metals grade.

6.4 ICP quality standard stock solutions are available from Inorganic Ventures, Spex, Plasma Pure, Ultra, Environmental Express, or equivalent.

6.5 Calibration Standards. These can be made up by diluting the stock solutions to the appropriate concentrations. The calibration standards should be prepared using the same type of acid (s) and at approximately the same concentration as will result in the samples following sample preparation.

6.5.1 For calibration and quantitation an internal standard (Yttrium/Indium) is used to limit nebulization problems. If it is known that the samples contain a significantly different acid matrix, the samples must be diluted so that they are in a similar matrix to the curve. All sample results are referenced to the initial calibration blank (ICB) Internal Standard counts. The criteria is 60-125 percent of the initial calibration blank (ICB) counts. If the internal standard counts fall outside these criteria matrix effects must be suspected and the sample diluted until it meets the criteria or footnoted in LIMS as suspected matrix interference.

6.5.2 Standards must be prepared so that there is minimal spectral interference between analytes.

Note: All Ag stock and intermediate solutions must be stored away from direct sunlight.

6.6 Analytical Quality Control Solutions.

All of the solutions below are prepared by adding either mixed or single element metals solutions to a solution prepared using the same type of acid (s) and at approximately the same concentration as will result in the samples following sample preparation.

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6.6.1 Blank (Calibration, ICB, CCB)

This reagent blank contains Nitric Acid at 3 percent and Hydrochloric Acid at 5 percent.

6.6.2 Initial Calibration Verification solution.

This standard solution must be made from a different source than the calibration curve. The concentrations for each element must be within the range of the calibration curve and should be approximately at the midpoint of the curve. This solution is used to verify the accuracy of the initial calibration. Levels for the ICV standard are shown in Table 4.

6.6.3 Continuing Calibration Verification solution.

The metals concentrations for this standard should be at approximately the mid point of the calibration curve for each element. This standard should be prepared from the same source that is used for the calibration curve. Levels for the CCV standard are shown in Table 5.

6.6.4 Interference Element Check Solutions.

These solutions must be analyzed to check the interfering element correction factors (IEC's) on the ICP instruments. Refer to section 17.0 of this SOP for further information regarding generation of IEC's.

6.6.4.1 ICSA Solution.

The ICSA solution contains only the interfering elements. Levels for the ICSA are shown in Table 9.

6.6.4.2 ICSAB Solution.

The ICSAB solution contains both the interferents and the analytes of interest. Levels for the ICSAB are shown in Table 10.

6.6.4.3 Single element interference check solutions

Prepared as single solutions. Levels for the single element interference solutions are shown in Table 11.

6.7 CRIA Standard Solution (Also referred to as LLCCV)

The CRIA standard contains the elements of interest at levels equal to Accutest Southeast quantitation limits (RL). Please refer to Table 6 for list of elements of interest and concentration levels for the CRIA. If special client reporting limits are requested, then low checks corresponding to those reporting limits must also be analyzed.

6.8 Matrix Spike, Matrix Spike duplicate, and Spike Blank Solution.

This solution is prepared by adding either mixed or single element metals solutions to a solution containing 3 percent nitric acid and 5 percent hydrochloric acid and diluting to a fixed

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final volume with this acid mixture. Spiking solution (s) must be added to the spike blank, matrix spike, and the matrix spike duplicate prior to digestion. Levels for the MS and MSD and Spike Blank standard are shown in Table 7.

6.9 Liquid Argon or Argon Gas. (99.999% purity)

7.0 ANALYTICAL PROCEDURE

Note: Please refer to section 8 of this SOP for further detail on quality control standards. Please refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

7.1 General procedure on how to operate the Thermo 6500 is described below. Refer to the Thermo 6500 operation manual for further details.

7.2 Before starting up the instrument, make sure that the pump tubing is in good condition, the torch assembly, the nebulizer, and the spray chamber are clean, the dehumidifier (if used) is filled with DI water up to the level between Minimum and Maximum, and that there are no leaks in the torch area.

7.3 Turn on the recirculating cooler. Verify that the argon is turned on and there is enough for the entire days analytical run.

7.4 Tighten the pump platens and engage the peristaltic pump. Make sure sample and internal standard solutions are flowing smoothly.

7.5 Put a new solution of acid rinse into the rinse reservoir. The composition of the rinse solution may be periodically changed to minimize sample introduction problems and sample carryover. If internal standard is being used, make sure that sufficient amount of internal standard is prepared for the entire analytical run.

7.6 Start up the instrument following the sequence show below.

7.6.1 Double click the **iTEVA Control Center** Icon on the desktop. Type ***admin*** in User Name field, and then click **OK**.

7.6.2 Once the iTEVA Control Center window is opened, click on **Plasma** Icon at status bar area. Then click on **Instrument Status** to check the interlock indicators (torch compartment, purge gas supply, plasma gas supply, water flow and exhaust should be in green; drain flow and busy should be in gray) and the Optics Temperature. (It should be around 38°C.) Click on the Close box.

7.6.3 Click **Plasma On**. When the plasma is on, click close. Let the instrument warm up for 15 to 20 minutes before starting the analysis. New tubing may take an hour to stabilize.

7.7 Torch Alignment and Auto Peak

7.7.1 If the torch has been cleaned, then the torch alignment procedure must be performed.

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7.7.2 Open the method and then click on **Sequence** tab, then click on **List View** Icon until you reach rack display.

7.7.3 Go to S-6 position (you can assign any position in the rack for torch alignment), then right click to select **Go** to empty sample S:6. (Now, the auto sampler tip moves from Rinse to this position).

7.7.4 Click on **Analysis** tab, then select **Torch Alignment** from Instrument drop down menu. There will be a pop up dialog box present. Click **Run**. Then there will be another dialog pop up box (This is a reminder for Torch Alignment Solution (2 ppm Zn)), click **Ok**. Now, the instrument is initializing an automated torch alignment. It takes about 7 minutes to complete this step. Progress is indicated in the progress bar.

7.7.5 After torch alignment is complete, click **Close**. Click on **Sequence** tab, then followed by **List View** Icon.

7.7.6 Go to Rinse position at rack display, right click to select Go to rinse and let it rinse for approximately 5 minutes.

7.7.7 Perform Auto Peak

7.7.8 It is recommended that the Auto Peak Adjust procedure be performed daily prior to calibration. A standard that contains all of the lines of interest is used and the system automatically makes the appropriate fine adjustments. (High standard solution should be used for this process.)

7.7.9 Click **Sequence** tab, then click on **List View** Icon until the rack is displayed.

7.7.10 Go to S-5 position (you can assign any position in the rack for auto peak adjust), then right click to select **Go** to empty sample S:5. (Now, the auto sampler tip moves from the Rinse position to this position). Click on **Analysis** tab. All elements result is shown in the display area. From Instrument drop down menu, select **Perform Auto Peak**. There will be a pop up dialog box present. Highlight “All Elements”, and then click **Run**. Then there will another pop up dialog box (This is a reminder for Auto Peak Solution), click **Ok**. Now, the instrument is performing auto peak adjust. It takes about 5 minutes to complete this process. The Auto Peak dialog box will show a green check mark in front of “All Elements”, which indicates Auto Peak is complete.

7.8 Open the method and start up the run.

7.8.1 Click on **Analyst** Icon at the workspace. Go to the method and choose Open from the drop down menu. Select the method with the latest revision number.

7.8.2 Go to **Method** tab at the bottom of left hand corner to click on **Automated Output** at the workspace area. Type a filename in Filename field in the data display area (i.e. : SA101010M1, starts with SA, then followed by MM-DD-YY, then M1; M1 indicates the first analytical run for that day, then followed by M2, M3 and so on for the second and third runs.) Click on **Apply To All Sample Types**.

7.8.3 Click on **Sequence** tab at the bottom of left hand corner. From Auto Session drop down menu bar, click on **New Auto sampler** to create a sequence. This will pop up a

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dialog box, then click on **New** and fill in number of samples (i.e.: 100) in the Number of Samples field and the sample I.D. (leave this field empty) in Sample Name field. Type a sequence name (i.e. : SEQ101010M1, starts with SEQ, then MM-DD-YY, then M1; M1 indicates the first analytical run for that day, then followed by M2, M3 and so on for the second and third runs) in the Sequence Name field. Click Ok, then put in "0" as settle time between between sequences, and click **Ok**.

7.8.4 Right click on **Untitled** (Cetac ASX-520 Enviro 5 Named Rack is the rack that is currently used) at the workspace area, click on **Auto-Locate All** to locate all sample positions.

7.8.5 Double click on **Untitled** again, then click on the sequence name (i.e. : SEQ101010M1), on the data display area, type the sequence in Samplename column, dilution factor (if needed) in CorrFact column, check the box in front of Check column, and select an appropriate check table.

7.8.6 Once done with creating sequence, go to **Method** drop down menu and save all changes as **Save As**. There will be a Save a Method dialog box present, go to the save option to check on "Overwrite Method and bump revision number" box, and then click **Ok**.

7.8.7 Go to Sequence tab, click on List View Icon from tool bar, then click on Connect Autosampler to PC and Initialize Icon.

7.8.8 See table 8 for a typical run sequence.

7.9 Calibrate the instrument as outlined below. See table 3 for calibration standards concentrations. This calibration procedure is done a minimum of once every 24 hours. The calibration standards may be included in the auto sampler program or they may be run manually from the **Calibrate Instrument (graduated cylinder)** icon located on the Analyst tab. All curves must be determined from a linear calibration prepared in the normal manner using the established analytical procedure for the instrument. Refer to instrument manual for further detail. Unless otherwise approved, the calibration curve must be determined by a blank and a series of three standards representing the elements of interest. Three exposures will be used with a percent relative standard deviation of less than 5 percent. The resulting correlation coefficient must be $\geq$0.998. If the calibration curves do not meet these criteria, analysis must be terminated, the problem corrected, and instrument re-calibrated. Correlation coefficients, slopes, and y-intercepts for each wavelength are printed and included in each analytical data package.

7.10 After the instrument is properly calibrated, begin by reanalyzing the high standard(s) for each element. The standards can be combined into one solution for this analysis. The analyzed value must be within 5 percent of the true value or that element must be re-calibrated. The High Standard Check shall be used for 200.7 only. After the high standards are analyzed, the ICV check standard shall be run. For the ICV, all elements to be reported must be within 5 percent of the true value for 200.7 and 10 percent of the true value for 6010C. If the ICV fails, analysis shall be terminated, problem corrected, and the instrument re-calibrated.

7.11 After analyzing the ICV, the ICB must be analyzed. The results of the ICB must be less than one half the reporting limit. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a "B" or "V" qualifier, or

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raising the reporting limit for all samples to greater than two times the background concentration.

7.12 Before analyzing any real samples the CRIA (also referred to as LLCCV) must be analyzed. The CRIA contains elements of interest at the reporting limit. The CRIA will be analyzed at the beginning and end of each analytical run. For all elements the results must be within 20 percent of the true value for client specific reporting limits (CRIA Requirement). For all others a 30 percent criterion will be applied. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits (CRIA Requirement). If the initial CRIA fails no samples associated with the failing CRIA can be reported, and the CRIA should be reanalyzed for the failing elements. If the closing CRIA fails the criteria, the samples associated with the CRIA shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the CRIA, or qualifying the results in LIMS.

7.13 Before analyzing any real samples, the interference check standards (ICSA, ICSAB) must be analyzed. For all spiked elements, the analyzed results must be within 20 percent of the true value. For non-spiked elements, the interfering element solutions must be $\pm$ the absolute value of the reporting limit for each element. Also, on an as needed basis (i.e.: instrument repair), analyze the single element interference check solutions (SIC). The same criteria as outlined above apply. If the ICSA and/or the ICSAB fall outside this criterion the problem must be corrected and the instrument re-calibrated or data footnoted in LIMS system. If the closing ICSA/ICSAB fails the criteria, the samples associated with the ICSA/ICSAB shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the ICSA/ICSAB, or qualifying the results in LIMS. Refer to section 17.0 of this SOP for Interfering Element Correction (IEC) procedure.

7.14 After the initial analytical quality control has been analyzed, the samples and the preparation batch matrix quality control shall be analyzed. Each sample analysis must be a minimum of 3 readings using at least a 5 second integration time. Between each sample, flush the nebulizer and the solution uptake system with a blank rinse solution for at least 60 seconds or for the required period of time to ensure that analyte memory effects are not occurring.

7.15 Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value for method 6010C and 5 percent for method 200.7 (for the initial CCV (ICCV)), the CCV shall be reanalyzed to confirm the initial value. If the CCV is not within criteria after the reanalysis, no samples can be reported in the area bracketed by the failing CCV. Immediately following the analysis of the CCV the CCB shall be analyzed. The results of the CCB must be less than one half the reporting limit for all elements. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit for all samples to greater than two times the background concentration.

7.16 One sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution (SDL) must be prepared. For the serial dilution, a 1:5 dilution must be made on the sample. The results of the 1:5 dilution shall agree within 10 percent of the true value as long as the sample and the dilution result are greater than 10 times the method detection limit or greater than 50 times the IDL. If the results are outside these criteria then matrix interference should be suspected and the proper footnote entered into LIMS. A post digestion spike (PDS) must be performed if the SDL fails. The PDS must

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recover within ± 20 percent for method SW846-6010C and ± 15 percent for method EPA 200.7. If the PDS is outside these limits then matrix interference must be suspected and the proper footnote entered into LIMS.

7.17 The upper limit of quantitation may exceed the highest concentration calibration point and can be defined as the "linear dynamic" range. Sample results above the linear dynamic range shall be diluted under the linear dynamic range and reanalyzed. Samples following a sample with high concentrations of analyte (s) must be examined for possible carryover. Verification may be done by rinsing the lines with an acid solution and then reanalyzing the sample. A limit check table is built into the autosampler file so that samples exceeding the standardization range are flagged on the raw data.

7.18 After the instrument is optimized and all initial QC has been run, click on **Run Auto-Session** Icon to start the analytical run sequence.

7.18.1 If you need to add or delete samples once the run is started, follow the steps shown below.

7.18.2 Click on **Sequence** tab, then click on **List View** Icon at the tool bar. There is the sequence table shown on the display area.

7.18.3 Click on **Add Samples** Icon. This will pop up a dialog box, and then fill in number of samples that need to be added. Click **Ok**. By doing this, samples will be added to the end of the current sequence without a rack location.

7.18.4 On the Samplename column type in the sample I.D., correction factors, and check tables. **Click on Auto Locate All.**

7.18.5 The added samples will be analyzed at the end of the original sequence run order unless they are assigned a different run order.

7.18.6 Deleting Samples

7.18.7 Click on **Sequence** tab, and then click on **List View** Icon under the sequence display area.

7.18.8 Highlight all samples that need to be deleted and then click on the **Delete Samples** icon.

7.19 When the analysis is completed export the data to LIMS following the procedure outlined below.

7.19.1 Double click on **ePrint** Icon on desktop. There will be a **LEADTOOLS ePRINT** pop up box, click on **Finish Jobs** and **OK** boxes.

7.19.2 Double click the **PDF** Icon on the desktop; the PDF file will be present as Document_#. Right click on that file, select **rename** to change the filename to an assigned analytical run I.D. (i.e.: MA9000). This is the raw data file for MA9000.

7.19.3 Drop the raw data to the **LIMS Data Drop** icon located on the desktop.

7.19.4 By completing the above steps, the raw data (i.e.: MA9000) can be viewed and/or printed from the Raw Data Search function.

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7.19.5 Go to **Analysis** tab, right click on sample header, and select export all samples. A pop up dialog box will come up, type in the analytical run I.D. (i.e.: SA101010M1) and click **Ok**. Go to **Lims Export** folder located on the desktop, right click on analytical run and change extension from .TXT to .ICP. Open the analytical file and make any necessary changes, such as deleting any samples that need to be re-run on dilution. **Save** the file. Drop the data file to the **LIMS Data Drop** icon located on the desktop. This will then send the export file to LIMS for review.

7.20 The data can be evaluated by running an automated data evaluation program, which will help to generate quality control summary pages. Each run must be evaluated as quickly as possible to make sure that all required quality control has been analyzed. With each data package include: cover sheet, copies of all prep sheets, autosampler run sequence, dilution sheets, and raw data. Label each folder with MA#, instrument run I.D., instrument used, and date.

7.21 At the end of the analysis day the ICP must be shutdown using the following sequence.

7.21.1 Place the auto sampler tip in the rinse cup and rinse in a mixed solution of approximately 5 percent nitric acid and 5 percent hydrochloric acid for 10 minutes and then in DI water for 20 minutes.

7.21.2 Turn off the plasma by clicking on the **Plasma** Icon and then by clicking **Plasma Off.**

7.21.3 Close all iTeva programs/windows.

7.21.4 Release the tension on the sample pump platens.

7.21.5 Turn off recirculating chiller.

8.0 QUALITY CONTROL

This section outlines the QA/QC operations necessary to satisfy the analytical requirements for method SW846 6010C. Please refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements. Check with the area supervisor or lab manager for any non compliant quality control for further information.

8.1 High Standard Check.

After the instrument is properly calibrated, the high standard(s) shall be reanalyzed for each element. The analyzed value must be within 5 percent of the true value. If the High Standard falls outside this criteria analysis shall be terminated, problem corrected, and the instrument re-calibrated.

Note: High Standard Check is for method 200.7 only. The standards can be combined into one solution for this analysis.

8.2 Initial Calibration Verification Standard (ICV).

After each calibration, a standard from a different source than the calibration standard shall be analyzed. For the ICV, all elements to be reported must be within 10 percent of the true value for 6010C and within 5 percent for 200.7. If the ICV is outside these

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criteria then the analysis must be terminated, problem corrected, and the instrument re-calibrated.

8.3 Continuing Calibration Blank/Initial Calibration Blank.

Analyze the Initial calibration blank solution at the beginning of each run and the continuing calibration blank after every tenth sample and at the end of the sample run. The ICB/CCB must be less than one half the reporting limit for each element. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit for all samples to greater than two times the background concentration.

8.4 Low Standard Check (CRIA).

The CRIA (also referred to as LLCCV) contains elements of interest at the reporting limit. The CRIA will be analyzed at the beginning and end of each analytical run. For all elements the results must be within 20 percent of the true value for client specific reporting limits (CRIA Requirement). For all others a 30 percent criterion will be applied. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits (CRIA Requirement). If the initial CRIA fails no samples associated with the failing CRIA can be reported, and the CRIA should be reanalyzed for the failing elements. If the closing CRIA fails the criteria, the samples associated with the CRIA shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the CRIA, or qualifying the results in LIMS.

8.5 ICSA and ICSAB and Single Element Interference Solutions

Analyze the ICSA and ICSAB at the beginning and end of each run following the analysis of the CRIA. Also, on an as needed basis (i.e.: instrument repair), analyze the single element interference check solutions (SIC). For all spiked elements, the analyzed results must be within 20 percent of the true value. For non-spiked elements, the interfering element solutions must be $\pm$ the absolute value of the reporting limit for each element. If the ICSA and/or the ICSAB fall outside this criterion the problem must be corrected and the instrument re-calibrated or data footnoted in LIMS system. If the closing ICSA/ICSAB fails the criteria, the samples associated with the ICSA/ICSAB shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the ICSA/ICSAB, or qualifying the results in LIMS. Refer to section 17.0 of this SOP for Interfering Element Correction (IEC) procedure.

8.6 Continuing Calibration Verification.

Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value for method 6010C and 5 percent for method 200.7 (for the initial CCV (ICCV)) the CCV must be reanalyzed to confirm the initial value. If the CCV is not within criteria after reanalysis no samples can be reported in the area bracketed by the failing CCV.

8.7 Method Blank.

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The laboratory must digest and analyze a method blank with each batch of samples. The method blank must contain elements at less than one half the reporting limit for each element. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit. Samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-digesting and reanalyzing the samples, qualifying the results with a "B" or "V" qualifier, or raising the reporting limit to greater than two times the background concentration,

8.8 Blank Spike Sample.

The laboratory must digest and analyze a spike blank sample with each batch of samples. Blank Spikes must be within 20 percent of the true value for method SW846-6010C and within 15 percent for method EPA 200.7. If the lab control is outside of the control limits for a reportable element, all samples must be re-digested and reanalyzed for that element. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results may be reported with no flag. For solid standard reference materials (SRMs) $\pm$ 20 percent accuracy may not be achievable and the manufacturer's established acceptance criterion should be used for all soil SRMs.

8.9 Matrix Spike and Matrix Spike Duplicate Recovery.

The laboratory must digest and analyze a matrix spike and matrix spike duplicate with each batch of samples. The matrix spike recovery is calculated as shown below and must be within 20 percent of the true value for method SW846-6010C and within 30 percent for method EPA 200.7. If a matrix spike is out of control, then the results must be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and must be footnoted to that effect.

Note: Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{Amount Spiked}} \times 100 = \text{matrix spike recovery}$$

8.10 Matrix Duplicate/Matrix Spike Duplicate Relative Percent Difference.

The laboratory must digest a duplicate with each batch of samples. The relative percent difference (RPD) between the duplicate and the sample must be assessed and must be $\leq$ 20 percent for sample results at or above the reporting limit. If the RPD is outside the 20 percent criteria the results must be qualified in LIMS. RPD's are also calculated in LIMS for sample results below the reporting limit. RPD's outside the 20 percent criteria are not considered failing and LIMS automatically footnotes these as "RPD acceptable due to low duplicate and sample concentrations."

Note: Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

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8.11 Serial Dilution Analysis and Post Digestion Spike.

For one sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution must be prepared. For the serial dilution, a 1:5 dilution must be made on the sample. The results of the 1:5 dilution must agree within 10 percent of the true value as long as the sample and the dilution result are greater than 10 times the method detection limit and/or greater than 50 times the IDL. If the dilution does not agree, then the sample must be post digestion spiked (PDS) at a level no less than 10 times but no greater than 100 times the MDL concentration. The PDS must recover within ± 20 percent for method SW846-6010C and ± 15 percent for method EPA 200.7. If the PDS is outside these limits then matrix interference must be suspected and the proper footnote entered into LIMS.

$$\frac{\text{(Sample Result - Serial Dil. Result)}}{\text{Sample Result}} \times 100 = \text{Serial Dilution RPD}$$

8.12 Linear Calibration ranges.

The upper limit of the linear calibration ranges must be established for all elements by determining the signal responses from a minimum of three concentration standards, one of which is close to the upper limit of the linear range. The linear calibration range, which may be used for the analysis of samples must be judged by the analyst from the resulting data. The upper range limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Linear calibration ranges must be determined whenever there is a significant change in instrument response or at a minimum, every 6 months.

8.13 Sample RSD

For samples containing levels of elements greater than five times the reporting limits, the relative standard deviation for the replicates should be less than 5%. If not, reanalyze the sample. If upon reanalysis, the RSD's are acceptable then report the data from the reanalysis. If RSD's are not acceptable upon reanalysis, then the results for that element should be footnoted that there are possible analytical problems and/or matrix interference indicated by a high RSD between replicates.

8.14 Interelement Spectral Interference Correction Validity

For the interelement spectral interference corrections to remain valid during sample analysis, the interferent concentration must not exceed its linear range. If the interferent concentration exceeds its linear range or its correction factor is big enough to affect the element of interest even at lower concentrations, sample dilution with reagent blank and reanalysis is required. In these circumstances, analyte dilution limits are raised by an amount equivalent to the dilution factor.

8.15 Internal Standard (Yttrium/Indium)

For any readings where the internal standard is outside of the range 60-125 percent of the internal standard level in the reference standard (Initial Calibration Blank), then the sample must be diluted until the internal standard is within range and all sample results must be footnoted in LIMS.

8.16 MSA (Method of Standard Additions)

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Accutest Southeast uses the internal standard technique as an alternative to the MSA per SW846-6010C section 4.4.2. However, in certain circumstances MSA may be needed by some project specific requirements. Accutest Southeast may perform an MSA when sample matrix interference is confirmed through the post digestion spike process or may qualify the results in LIMS. Accutest Southeast will use a single addition method as described in SW846-7000B.

9.0 GLASSWARE CLEANING

All glassware must be washed with soap and tap water and then rinsed with 5 percent nitric acid. It must then be rinsed at least 3 times with DI water. Refer to SOP GN196, current revision for further information regarding glassware cleaning.

10.0 DOCUMENTATION REQUIREMENTS

Refer to the Laboratory Quality Assurance Manual for documentation requirements. All raw data is printed to .PDF format and archived to a backup server for long term storage.

11.0 SAFETY

The analyst must follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor. Follow proper safety precautions when working with gas cylinders.

12.0 CALCULATIONS

For water samples, the following calculations must be used. Refer to the QC section for the calculations to be used for the QC samples.

Original sample concentration of metal (ug/l) =

$$\frac{\text{(conc. in the digestate (ug/l)) x (final digestate volume (ml))}}{\text{(initial sample volume (ml))}}$$

For soil samples, the following calculations must be used.

Concentration of the metal in the dry sample (mg/kg) =

$$\frac{\text{(conc. in the digestate (mg/l) x final digestate volume(L))}}{\text{(sample wt. (kg)) x (\% solids/100)}}$$

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13.0 INSTRUMENT MAINTENANCE

Recommended periodic maintenance includes the items outlined below. All maintenance must be recorded in the instrument maintenance log.

13.1 Change the pump tubing as needed.

13.2 Clean the filter on the recirculating pump approximately once a month and dust off the power supply vents as needed.

13.3 Clean or replace the nebulizer, torch assembly, and injector tube as needed.

13.4 Change the sampler tip as needed.

13.5 Clean the recirculating pump lines and internal sock filter every 3 months or as needed.

13.6 Clean the radial view quartz surface weekly or more often if needed.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 14.2.

14.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

15.0 GENERIC DEFINITIONS

15.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours which ever comes first.

15.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

15.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. A CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

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15.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

15.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the reporting level.

15.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

15.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the performance of a method in a given sample matrix.

15.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and performance of a method in a given sample matrix.

15.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

15.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

15.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

16.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Statistical control limits are stored in the LIMS for QA purposes only. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

17.0 GENERATION OF INTERFERING ELEMENT CORRECTION FACTORS

17.1 It is recommended that all IEC's be verified and updated approximately every 6 months or whenever instrument conditions change significantly. It is also recommended that elements with frequent high concentrations or with large IEC's should be checked more frequently.

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17.2 Calculate the IEC correction factors and enter them into the method (refer to Thermo 6500 instrument manual). Calculate the correction factor using the equation shown below. This correction factor must be added to the correction factor already in place in the method for a given element.

$$\text{IEC} = \frac{\text{Concentration Result of the element with the interference}}{\text{Concentration result of the interfering element}}$$

17.3 Verify the new correction factors by reanalyzing the ICSA/ICSAB solutions and/or the SIC solutions or by reloading and recalculating the previously stored results. If the reanalysis is not within QC limits, make additional changes to the IEC factors and then re-verify both the individual and combined solution values.

17.4 Save and update the method.

17.5 Interfering element correction factors are saved as raw data along with the run printouts on a daily basis so that the IEC's for a given run are traceable.

TABLE 1: REPORTING LIMIT BY ELEMENT

	Water	Soil	TCLP
Analyte	**Reporting Limit (ug/L)**	**Reporting Limit (mg/kg)**	**Reporting Limit (mg/L)/MCL**
Tin	50	5	
Aluminum	200	20	
Antimony	5	1	
Arsenic	10	0.5	0.10 / 5.0
Barium	200	20	10 / 100
Beryllium	4	0.5	
Cadmium	5	0.4	0.05 / 1.0
Calcium	1000	500	
Chromium	10	1	0.10 / 5.0
Cobalt	50	5	
Copper	25	2.5	
Iron	300	10	
Lead	5	1	0.5 / 5.0
Magnesium	5000	500	
Manganese	15	1.5	
Nickel	40	4.0	
Potassium	5000	500	
Selenium	10	1	0.5 / 1.0
Silver	10	1	0.10 / 5.0
Sodium	5000	500	

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Thallium	10	1
Vanadium	50	5
Zinc	20	2
Molybdenum	50	2.5
Strontium	10	0.5
Titanium	10	0.5

TABLE 2. THERMO 6500 ANALYSIS LINES

Element	Wavelength
Al	396.1
As	189.042
Ca	317.933
Fe	259.9
Mg	279.078
Mn	257.610
Pb	220.353
Se	196.026
Tl	190.864
V	292.402
Ag	328.068
Ba	455.4
Be	313.042
Cd	226.502
Co	228.616
Cr	267.716
Cu	324.753
K	766.491
Na	589.5
Ni	231.604

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Sb	206.838
Zn	206.2
Mo	202.030
Sn	189.900
Sr	407.7
Ti	334.9

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TABLE 3: LOW, MID AND HIGH STANDARD LEVELS

Element	Low	Mid	High
	ug/l	ug/l	ug/l
Al	10000	40000	80000
As	500	2000	4000
Ca	10000	40000	80000
Fe	10000	40000	80000
Mg	10000	40000	80000
Mn	500	2000	4000
Pb	500	2000	4000
Se	500	2000	4000
Tl	500	2000	4000
V	500	2000	4000
Ag	62.5	250	500
Ba	500	2000	4000
Be	500	2000	4000
Cd	500	2000	4000
Co	500	2000	4000
Cr	500	2000	4000
Cu	500	2000	4000
K	10000	40000	80000
Na	10000	40000	80000
Ni	500	2000	4000
Sb	500	2000	4000
Zn	500	2000	4000
Mo	500	2000	4000
Sn	500	2000	4000
Sr	500	2000	4000
Ti	500	2000	4000

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TABLE 4: ICV STANDARD LEVELS

Element	Concentration
	ug/l
Al	40000
As	2000
Ca	40000
Fe	40000
Mg	40000
Mn	2000
Pb	2000
Se	2000
Tl	2000
V	2000
Ag	250
Ba	2000
Be	2000
Cd	2000
Co	2000
Cr	2000
Cu	2000
K	40000
Na	40000
Ni	2000
Sb	2000
Zn	2000
Mo	2000
Sn	2000
Sr	2000
Ti	2000

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TABLE 5: CCV STANDARD LEVELS

Element	Concentration
	ug/l
Al	40000
As	2000
Ca	40000
Fe	40000
Mg	40000
Mn	2000
Pb	2000
Se	2000
Tl	2000
V	2000
Ag	250
Ba	2000
Be	2000
Cd	2000
Co	2000
Cr	2000
Cu	2000
K	40000
Na	40000
Ni	2000
Sb	2000
Zn	2000
Mo	2000
Sn	2000
Sr	2000
Ti	2000

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TABLE 6: CRIA STANDARD LEVELS

Element	CRIA
	ug/l
Al	200
As	10
Ca	1000
Fe	300
Mg	5000
Mn	15
Pb	5
Se	5
Tl	10
V	50
Ag	10
Ba	200
Be	5
Cd	5
Co	50
Cr	10
Cu	25
K	5000
Na	5000
Ni	40
Sb	5
Zn	20
Mo	50
Sn	50
Sr	10
Ti	10

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TABLE 7: BLANK SPIKE, MATRIX SPIKE AND MATRIX SPIKE DUPLICATE LEVELS

Element	Concentration
	ug/l
Al	27000
As	2000
Ca	25000
Fe	26000
Mg	25000
Mn	500
Pb	500
Se	2000
Tl	2000
V	500
Ag	50
Ba	2000
Be	50
Cd	50
Co	500
Cr	200
Cu	250
K	25000
Na	25000
Ni	500
Sb	500
Zn	500
Mo	500
Sn	500
Sr	500
Ti	500

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TABLE 8: TYPICAL RUN SEQUENCE

BLANK
MID
HIGH
HIGH STD
ICV
ICB
CRIA
ICSA
ICSAB
CCV
CCB
MB
SB
SAMPLE1
DUPLICATE
SERIAL DILUTION
MATRIX SPIKE
MATRIX SPIKE DUPLICATE
POST DIGESTION SPIKE
SAMPLE2
SAMPLE3
CCV
CCB
SAMPLE4
SAMPLE5
SAMPLE6
SAMPLE7
SAMPLE8
SAMPLE9
SAMPLE10
SAMPLE11
SAMPLE12
SAMPLE13
CRIA
ICSA
ICSAB
CCV
CCB
ETC.

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TABLE 9: ICSA SOLUTION LEVELS

Element	Concentration
	mg/l
Al	500
As	0
Ca	500
Fe	200
Mg	500
Mn	0
Pb	0
Se	0
Tl	0
V	0
Ag	0
Ba	0
Be	0
Cd	0
Co	0
Cr	0
Cu	0
K	0
Na	0
Ni	0
Sb	0
Zn	0
Mo	0
Sn	0
Sr	0
Ti	0

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TABLE 10: ICSAB SOLUTION LEVELS

Element	Concentration
	mg/l
Al	500
As	1.0
Ca	500
Fe	200
Mg	500
Mn	0.5
Pb	1.0
Se	1.0
Tl	1.0
V	0.5
Ag	1.0
Ba	0.5
Be	0.5
Cd	1.0
Co	0.5
Cr	0.5
Cu	0.5
K	0
Na	0
Ni	1.0
Sb	1.0
Zn	1.0
Mo	1.0
Sn	1.0
Sr	1.0
Ti	1.0

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TABLE 11: SINGLE ELEMENT INTERFERENCE CHECK SOLUTION (SIC) LEVELS

Element	Concentration
	mg/l
Al	500
As	0
Ca	500
Fe	200
Mg	500
Mn	0
Pb	0
Se	0
Tl	0
V	0
Ag	0
Ba	0
Be	0
Cd	0
Co	0
Cr	0
Cu	0
K	0
Na	0
Ni	0
Sb	0
Zn	0
Mo	0
Sn	0
Si	50
Sr	0
Ti	0

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DIGESTION OF WATER SAMPLES FOR METALS ANALYSIS BY ICP

Prepared by: Dave Metzgar Date: 09/04/2013

Reviewed by: Svetlana Izosimova Date: 09/13/2013

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

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Effective 7 days after "*" date

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TEST NAME: DIGESTION OF WATER SAMPLES FOR METALS ANALYSIS BY ICP

METHOD REFERENCE: SW846 3010A, EPA 200.7, SM 3030C

DEPARTMENT: METALS

REPORTING LIMIT: NOT APPLICABLE

REVISIONS: Section 2.0: Added detail about pH
Section 6.8: First sentence rewritten
Section 7.3: Changed CPI to Environmental Express
Section 1 and 8.7 – SM 3030 revision corrected

1.0 SCOPE AND APPLICATION, SUMMARY

This method is applicable for the digestion of aqueous samples, TCLP extracts and wastes that contain small amounts of suspended solids. After digestion, the samples can be analyzed by ICP. The digestion methods described in this SOP are based upon SW846 method 3010A, EPA 200.7, and Standard Methods 3030C water digestion methods.

Reduced volume version of method 200.7, March 1983 is used for the 200.7 digestion procedure. This approach that uses the same reagents and molar ratios is acceptable by the regulatory agents provided it meets the quality control and performance requirements stated in the method.

Acid Extractable Metals by method SM3030C-2004 modified volumes.

2.0 PRESERVATION AND BOTTLEWARE

All samples should be preserved with nitric acid to a pH of <2 at the time of collection. All sample pH are checked in sample receiving and within the metals department. Samples that are received with a pH >2 must be preserved to pH <2 and held for 24 hours prior to metals digestion to dissolve any metals that absorb to the container walls. Refer to SOP SAM101, current revision for further instruction. Final pH of TCLP extracts are checked and recorded in Accutest Southeast Extractions Department. Please refer to TCLP (1311) fluid determination logbook and SPLP (1312) fluid determination logbook for further information. TCLP extracts received from Accutest Southeast Extractions Department are prepared as soon as possible, no longer than 24 hours from time of receipt. If precipitation is observed during the sample preparation process the sample(s) are immediately re-prepped on dilution until no precipitation is observed. Samples received for dissolved metals analysis should be filtered and preserved to pH<2 as soon as possible. Refer to Accutest Southeast Sample Filtration Logbook for further information. All bottleware used by Accutest Southeast is tested for cleanliness prior to shipping to clients. Analysis results must be < ½ RL to be acceptable. Refer to SOP SAM104, current revision for further instruction.

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3.0 HOLDING TIME AND STORAGE

All samples should be digested and analyzed within 6 months of the time collection.

Samples being digested by method 3030C must be prepared within 72 hours of collection.

Aqueous samples do not require refrigeration.

4.0 REPORTING and METHOD DETECTION LIMITS

See analytical SOP MET100, current revision for further information.

5.0 INTERFERENCE

Organic substances in a matrix may cause interference if the sample is not digested rigorously enough. In addition, high levels of acids in the final digestate may cause interference in the analysis. This interference can be avoided by choosing the appropriate digestion method and by bringing the sample to an appropriate final volume. For a discussion of other interference, refer to specific analytical methods.

6.0 APPARATUS

The apparatus needed for this digestion procedure are listed below.

6.1 Automatic repipettor(s)

6.2 Fisher Brand 0.45 micron (um) filter or equivalent. Filter lots are checked for cleanliness through the Method Blank process. All Method Blank analytical results must be <1/2 RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated filters must be re-filtered through acceptable filters.

6.3 Environmental Express watch glasses or equivalent.

6.4 Thermometer(s)- capable of measuring a temperature of at least 125^0C, and checked against NIST traceable thermometers. Refer to SOP QA002, current revision for further information.

6.5 Environmental Express Hot Block or equivalent capable of maintaining a temperature of 90-95°C.

6.6 Environmental Express digestion vessels or equivalent, 50ml capacity. Each Lot of digestion tubes comes with a Certificate of Analysis which demonstrates

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cleanliness as well as volume accuracy. Please refer to Digestion Tube Certificate Logbook for further information. Tube Lots are also checked through the Method Blank process. All Method Blank analytical results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Re-digestion is required for all samples prepared with the contaminated tube lot.

6.7 Fisher Brand disposable 10 ml syringes or equivalent. Syringe lots are checked for cleanliness through the Method Blank process. All Method Blank results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated syringes must be re-filtered through acceptable syringes.

6.8 Eppendorf Pipette (s) - Pipette (s) are checked for accuracy and to ensure they are in good working condition. Volumes are checked at 100% of maximum volume, the 50% (mid-range) and between 10% and 25% at the low range, whichever constitutes most frequently used volume for a particular pipette. Pipettes are checked within the metals department approximately once per week and stored electronically in the “Eppendorf Calibration Log”. Refer to SOP QA006, current revision for further information regarding pipette calibration.

6.9 Class A volumetric flask (s)

6.10 Class A volumetric pipette (s)

6.11 Class A graduated cylinder (s)

7.0 REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Refer to Acid Certificate of Analysis logbook for Certificates of Analysis and compliance with the specifications of the grade listed. Accutest Laboratories produces DI water to the specifications for the ASTM Type II standard designation based on system manufacturer’s performance specifications. The DI water is used exclusively for laboratory purposes. Refer to SOP QA037, current revision for more information regarding testing and monitoring. De-ionized (DI) water should be used whenever water is required.

7.1 Hydrochloric acid. Fisher Trace metal grade or equivalent

7.2 Nitric acid. Fisher Trace metal grade or equivalent

7.3 Metals spiking solutions commercially purchased:

Environmental Express Multielement spiking solution or equivalent made with 5% HNO3 and a trace of HF.

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Inorganic Ventures 5000 mg/l Mineral solution or equivalent.

Prepared Metals standards:

100ppm Molybdenum, 100ppm Tin, 100ppm Strontium, and 100ppm Titanium spiking solution prepared as follows: Using a 10ml class A volumetric pipette, add 10mls of 1000ppm stock Molybdenum, 10mls of 1000ppm stock Tin, 10mls of 1000ppm stock Strontium, and 10mls of 1000ppm stock Titanium to a 100ml class A volumetric flask containing approximately 50mls of DI water and 3mls of concentrated Nitric acid and 5mls of concentrated HCL. Dilute to volume with DI water and mix well. This standard must be prepared every 6 months or before stock standard expiration date, whichever comes first. Refer to Metals Department Standard Prep Logbook for further information. Some of the information included in the logbook is as follows: standard name, elements in mix, manufacturer, lot number, parent expiration date, acid matrix, stock concentration, volume of standard added, total volume, final prepared concentration, prep date, initials, MET number, and prepared standard expiration date.

8.0 PROCEDURE

8.1 Shake sample vigorously to ensure thorough mixing. Measure out 50 ml of each sample into a labeled digestion vessel. The sample may be measured by using a Class A graduated cylinder or by using the calibrated digestion tube. Make sure that the sample identifications are accurately recorded on the digestion vessels and in the sample digestion log. In addition to the samples, a serial dilution (performed at the analytical bench), a post digestion spike (performed at the analytical bench), a matrix spike (MS), matrix spike duplicate (MSD), duplicate, blank spike and a method blank should be set up with each batch of 20 samples (10 samples for method 200.7). For the method blank and blank spike, 50 ml of DI water should be used. Refer to Table 1 for the spiking solution levels to use for each MS, MSD and blank spike.

8.1.1 When preparing TCLP samples use 5.0mls initial volume of extract and bring to a final volume of 50mls using DI water. Also prepare an additional leachate blank and leachate blank spike from the extraction fluid used for the samples. See section 8.8 and 8.9 of current METSOP 100 for acceptance criteria.

8.1.2 When preparing filtered samples for dissolved metals, an additional method blank must be prepared. This is performed to ensure there is no cross contamination from the filter media into the samples. The method blank must be filtered through the same filter media as the samples. See section 8.8 of current METSOP 100 for acceptance criteria.

8.2 Add 1.5 ml of concentrated nitric acid to all quality control and samples

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8.3 Pre heat the Hot Block to 90 to 95°C. Place the labeled digestion vessels into the heating apparatus. Heat the samples at a gentle reflux. Reduce the volume of each sample to approximately 5 ml. This should take approximately 2 hours. Allow samples to cool.

8.4 Add an additional 1.5-ml of concentrated nitric acid to all quality control and samples. Continue heating, adding additional acid if necessary, until the digestion is complete, generally indicated when the digestate is light in color or does not change in appearance with continued refluxing. Reduce volume to approximately 5mls. Allow samples to cool.

Note: If a client requires method 200.7 for the digestion, add 2.5ml of concentrated nitric acid instead of 1.5 ml in step 8.4. Continue heating the samples at a gentle reflux until the sample is completely digested. Signs of a complete digestion are if the digestate is light in color and/or if the appearance of the sample does not change with continued refluxing. More acid may be added as necessary to complete the digestion.

8.5 Add 2.5 ml of concentrated HCL to each sample and reflux for an additional 15 minutes. Allow samples to cool. Rinse digestion vessel walls with DI water.

Note: If a client requires method 200.7 for the digestion, add 7.5 ml of DI water along with the HCL.

8.6 Bring the samples to a final volume of 50.0 ml with DI water. If the sample contains particulate matter, it should be filtered (performed at analytical bench), along with the method blank and blank spike, through a 0.45um syringe filter before analysis. The sample is now ready for analysis by ICP.

8.7 Standard Method 3030C

Shake the sample vigorously to ensure thorough mixing. Transfer 50 ml to a labeled digestion vessel, and add 2.5 ml of 1 + 1 HCL. Heat 15 minutes on 90 to 95°C hotblock. Filter through a membrane filter and adjust filtrate volume to 50 ml with DI water. Sample is now ready for analysis. Please refer to Standard Methods 3030C-2004 for further instruction.

9.0 QC REQUIREMENTS

For each digestion batch of 20 samples (10 samples per batch for method 200.7), a serial dilution (performed at analytical bench), a post digestion spike (performed at the analytical bench), a matrix spike, a matrix spike duplicate, a duplicate, a blank spike (LCS), and a method blank should be prepared. Re-digestion is suggested for QC that does not meet the Accutest QC limits. The appropriate lab supervisor or lab manager will notify the analyst of samples that need re-digestion. Please refer to TABLE 1 in this

SOP for spiking volumes and concentrations. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

10.0 GLASSWARE CLEANING

All glassware should be washed with soap and tap water, rinsed with 5% nitric acid solution, and then rinsed at least 3 times with DI water. Refer to SOP GN196, current revision for further information regarding glassware cleaning.

11.0 DOCUMENTATION REQUIREMENTS

All digestion information should be documented in the Sample Digestion Logbook. The information required includes the sample identification (including the sample bottle number), the initial sample volume, and the final sample volume, the acids used (including lot number and manufacturer), the spiking solutions used, the digestion vessel lot number, the observed temperature, corrected temperature, the thermometer ID, analyst's signature, and the date of digestion. The analyst should write additional information such as unusual sample characteristics in the comment section.

12.0 SAFETY

The analyst should follow safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acid contacts any part of the body, flush with water and contact the supervisor immediately.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 13.2.

13.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

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14.0 GENERIC DEFINITIONS

14.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours which ever comes first.

14.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

14.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. A CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

14.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

14.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

14.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

14.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the performance of a method in a given sample matrix.

14.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and performance of a method in a given sample matrix.

14.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

14.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

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14.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

15.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Statistical control limits are stored in the LIMS for QA purposes only. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

16.0 HotBlock Maintenance

Clean surface area of hotblock periodically to prevent sample and reagent build up on the surface of the block. If the hotblock can not maintain a temperature between 90-95 degree C or the user experiences any other type of mechanical or electronic error a service representative will need to be contacted. Any hotblock that is not functioning properly must be tagged as “Out of Service”.

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Table 1: ICP Metals Spiking Levels

ELEMENT	INITIAL CONC (ppm)	VOLUME USED (ml)	FINAL CONC (mg/l)	FINAL VOL. (ml)
Ba	200	0.50	2.0	50
Be	5	0.50	.05	50
Cd	5	0.50	.05	50
Cr	20	0.50	.20	50
Cu	25	0.50	.25	50
Co	50	0.50	0.50	50
Mn	50	0.50	0.50	50
V	50	0.50	0.50	50
Zn	50	0.50	0.50	50
As	200	0.50	2.0	50
Se	200	0.50	2.0	50
Pb	50	0.50	0.50	50
Tl	200	0.50	2.0	50
Sb	50	0.50	0.50	50
Mo	100	0.25	0.50	50
Sn	100	0.25	0.50	50
Al	200/5000	0.5/0.25	27	50
Fe	200/5000	0.5/0.25	26	50
Mg	5000	0.25	25	50
Ca	5000	0.25	25	50
K	5000	0.25	25	50
Na	5000	0.25	25	50
Ag	5	0.50	0.05	50
Ni	50	0.50	0.50	50
Sr	100	0.25	0.50	50
Ti	100	0.25	0.50	50

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DIGESTION OF SOILS FOR ICP ANALYSIS

Prepared by: Dave Metzgar Date: 09/04/2013

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TEST NAME: DIGESTION OF SOILS FOR ICP ANALYSIS

METHOD REFERENCE: 3050B

DEPARTMENT: METALS

REPORTING LIMIT: Not applicable

REVISIONS: Section 6.4: Changed CPI to Environmental Express
Section 7.10: updated detail
Appendix A: page 12, Minimum sample size changed to 50 grams.

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is applicable for the digestion of sediments, soils, sludges and solid wastes. After digestion, the samples can be analyzed by ICP. This digestion method is based upon SW846 method 3050B.

1.2 An aliquot of a homogenized soil is digested with repeated additions of nitric acid and hydrogen peroxide. The volume is reduced to 5 ml and then hydrochloric acid is added and the sample is refluxed for 15 minutes. The sample is cooled to room temperature and diluted to 50 ml. If particulate matter is present, the sample is filtered.

2.0 PRESERVATION

All soils must be refrigerated at $\leq$ 6 ^{o}C. All bottleware used by Accutest Southeast is tested for cleanliness prior to shipping to clients. Analysis results must be < ½ RL to be acceptable. Please refer to SOP SAM104, current revision for further instruction.

3.0 HOLDING TIME

All samples should be digested and analyzed within 6 months of the time of collection.

4.0 INTEREFRENCES

Sludge and soil samples can contain diverse matrix types, which may contain a variety of interference. Spiked samples can be used to determine if this interference is adequately treated in the digestion process. For discussion of other interference, refer to specific analytical methods.

5.0 APPARATUS

The apparatus needed for this digestion procedure are listed below.

5.1 Automatic repipettor (s)

5.2 Fisher Brand 0.45 micron (um) filter or equivalent. Filter lots are checked for cleanliness through the Method Blank process. All Method Blank analytical results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated filters must be re-filtered through acceptable filters.

5.3 Top loader balance- capable of accurately weighing 0.01g. Refer to SOP QA005, current revision for balance calibration information.

5.4 Thermometer- capable of measuring to at least 125^0C and checked against NIST traceable thermometers. Refer to SOP QA002, current revision for further information.

5.5 Environmental Express Hot Block or equivalent capable of maintaining a temperature of 90-95°C.

5.6 Environmental Express digestion vessels or equivalent, 50ml capacity. Each Lot of digestion tubes comes with a Certificate of Analysis which demonstrates cleanliness as well as volume accuracy. Please refer to Digestion Tube Certificate Logbook for further information. Tube Lots are also checked through the Method Blank process. All Method Blank analytical results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Re-digestion is required for all samples prepared with the contaminated tube lot.

5.7 Fisher Brand disposable 10 ml syringes or equivalent. Syringe lots are checked for cleanliness through the Method Blank process. All Method Blank results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated syringes must be re-filtered through acceptable syringes.

5.8 Fisher Brand wooden spatulas or equivalent.

5.9 Eppendorf Pipette (s) - Pipette (s) are checked weekly for accuracy and to ensure they are in good working condition. Volumes are checked at 100% of maximum volume, the 50% (mid-range) and between 10% and 25% at the low range, whichever constitutes most frequently used volume for a particular pipette. Pipettes are checked within the metals department approximately once per week and stored electronically in the “Eppendorf Calibration Log”. Refer to SOP QA006, current revision for further information regarding pipette calibration.

5.10 Class A volumetric flask (s)

5.11 Class A volumetric pipette (s)

5.13 Teflon Chips

5.14 Solid Standard Reference Material (SRM) as required per project/client specific requirements.

6.0 REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Refer to Acid Certificate of Analysis logbook for Certificate of Analysis and compliance with specifications of the grade listed. De-ionized (DI) water should be used whenever water is required. Accutest Laboratories produces DI water to the specifications for the ASTM Type II standard designation based on the system manufacturer's performance specifications. The DI water is used exclusively for laboratory purposes. Refer to SOP QA037, current revision for more information regarding testing and monitoring.

6.1 Hydrochloric acid, Fisher Trace metal grade or equivalent

6.2 Nitric acid, Fisher Trace metal grade or equivalent

6.3 Hydrogen peroxide, reagent grade ,30%

6.4 Metals spiking solutions commercially purchased:

Environmental Express Multielement spiking solution or equivalent made with 5% HNO3 and a trace of HF.

Inorganic Ventures 5000 mg/l Mineral solution.

Prepared Metals Standards:

100ppm Molybdenum, 100ppm Tin, 100ppm Strontium, and 100ppm Titanium spiking solution prepared as follows: Using a 10ml class A volumetric pipette, add 10mls of 1000ppm stock Molybdenum, 10mls of 1000ppm stock Tin, 10mls of 1000ppm Strontium, and 10mls of 1000ppm Titanium to a 100ml class A volumetric flask containing approximately 50mls of DI water and 3mls of concentrated Nitric acid and 5mls of concentrated HCL. Dilute to volume with DI water and mix well. This standard must be prepared every 6 months or before stock standard expiration date, whichever comes first. Refer to Metals Standard Prep Logbook for further information. Some of the information included in the logbook is as follows: standard name, elements in mix, manufacturer, lot number, parent expiration date, acid matrix, stock concentration, volume of standard added, total volume, final prepared concentration, prep date, initials, MET number, and prepared standard expiration date.

7.0 PROCEDURE

7.1 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. The sample container must have enough room to move the matrix around with the wooden spatula. Mix the sample thoroughly using the wooden spatula. Make certain the entire sample is mixed well. The wooden spatula must reach the bottom of the original container and be able to be moved through the entire sample to ensure proper mixing. If the sample is packed tightly or matrix is dense and can not be efficiently moved around in the original jar, a secondary container such as a porcelain dish must be used. Remove the sample from the original container and place in the clean secondary container. While in the secondary container thoroughly mix sample around until appearing uniform in consistency. Upon completion the sample is re-packed into the original container. Refer to SOP QA034, current revision for more information on sample homogenization. Using a wooden spatula weigh out approximately 1.0 gram of a homogeneous sample on a top loading balance and place in the digestion vessel.

7.2 The sample identification must be accurately recorded on the digestion vessel and sample digestion log. In addition to the samples, a serial dilution (performed at the analytical bench), a post digestion spike (performed at the analytical bench), a matrix spike (MS), matrix spike duplicate (MSD), blank spike, duplicate (DUP) and a method blank should be set up with each batch of 20 samples. Refer to Table 1 for the spiking solution levels to use for each matrix spike, matrix spike duplicate, and blank spike. For the method blank and blank spike, 1.0 g of Teflon chips should be used. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

7.3 Add 2.5 ml of concentrated nitric acid to all quality control and samples.

7.4 Pre heat the Hot Block to 90 to 95°C. Place the labeled digestion vessels into the heating apparatus. Heat the samples at a gentle reflux for 10-15 minutes at 90 to 95°C. Allow the samples to cool.

7.5 Add an additional 2.5 ml of concentrated nitric acid to all quality control and samples. Heat the samples at a gentle reflux for an additional 30 minutes. Allow samples to cool.

7.5.1 If brown fumes are generated, which indicates oxidation of sample by HNO3, then repeat step 7.5 until no brown fumes are present.

7.5.2 Allow sample to evaporate to 5 ml without boiling or heat at 90 to 95°C without boiling for 2 hours. Do not allow sample to go to dryness.

7.6 Allow samples to cool. Add 2 ml of DI water and 3 ml of 30% hydrogen peroxide to each sample and reflux until effervescence subsides.

7.7 Continue to add 30% hydrogen peroxide in 1ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 10 mls of 30% hydrogen peroxide.

7.8 Allow sample to evaporate to 5 ml or heat at 90 to 95°C for 2 hours. Do not allow sample to go to dryness.

7.9 Allow samples to cool. Add 5 ml of concentrated HCl and reflux for an additional 15 minutes.

7.10 Allow the sample to cool. Dilute to final volume of 50 mls using DI water, cap and shake vessel. If particulate matter is present, uncap the vessel and filter using Fisher Brand disposable syringe and 0.45 micron (um) filter or equivalent. The method blank and blank spike for the filtered sample's prep group must be filtered as well. All samples are filtered at the analytical bench.

7.11 The sample is now ready for analysis by ICAP.

8.0 QC REQUIREMENTS:

For each digestion batch of 20 samples, a serial dilution (performed at the analytical bench), a post digestion spike (performed at the analytical bench), a matrix spike (MS), a matrix spike duplicate (MSD), a duplicate (DUP), a blank spike (LCS), and a method blank should be prepared. Re-digestion is suggested for QC that does not meet the Accutest QC limits. The appropriate lab supervisor or lab manager will notify the analyst of samples that need re-digestion. Please refer to TABLE 1 in this SOP for spiking volumes and concentrations. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

9.0 GLASSWARE CLEANING:

All glassware should be washed with soap and tap water and then soaked in a 5% nitric acid bath. It should then be rinsed at least 3 times with de-ionized water. Refer to SOP GN196, current revision for further information regarding glassware cleaning.

10.0 DOCUMENTATION REQUIREMENTS:

All digestion information should be completed in the Metals Digestion Log. The information required includes: the sample identification (including bottle number), the initial sample weight, the final sample volume, the acids (including the lot number and manufacturer), the spiking solutions used, the observed temperature, the corrected temperature, the thermometer ID, the digestion vessel lot number, the filter lot number, the Teflon chips lot number, analysts signature, and the digestion date. The analyst should write additional information such as unusual sample characteristics in the comment section.

11.0 SAFETY:

The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the uses of safety glasses and lab coats. In

addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact supervisor.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 12.2.

12.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

13.0 GENERIC DEFINITIONS

13.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours which ever comes first.

13.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

13.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. A CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

13.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

13.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

13.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

13.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

13.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and bias of a method in a given sample matrix.

13.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

13.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

13.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

14.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Statistical control limits are stored in the LIMS for QA purposes only. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

15.0 Hotblock Maintenance

Clean surface area of hotblock periodically to prevent sample and reagent build up on the surface of the block. If the hotblock can not maintain a temperature between 90-95 degree C or the user experiences any other type of mechanical or electronic error a service representative will need to be contacted. Any hotblock that is not functioning properly must be tagged as “Out of Service”.

TABLE 1: ICP METALS SPIKING LEVELS

ELEMENT	INITIAL CONC (ppm)	VOLUME USED (ml)	FINAL CONC (mg/l)	FINAL VOL. (ml)
Ba	200	0.50	2.0	50
Be	5	0.50	.05	50
Cd	5	0.50	.05	50
Cr	20	0.50	.20	50
Cu	25	0.50	.25	50
Co	50	0.50	0.50	50
Mn	50	0.50	0.50	50
V	50	0.50	0.50	50
Zn	50	0.50	0.50	50
As	200	0.50	2.0	50
Se	200	0.50	2.0	50
Pb	50	0.50	0.50	50
Tl	200	0.50	2.0	50
Sb	50	0.50	0.50	50
Mo	100	0.25	0.50	50
Sn	100	0.25	0.50	50
Al	200/5000	0.5/0.25	27	50
Fe	200/5000	0.5/0.25	26	50
Mg	5000	0.25	25	50
Ca	5000	0.25	25	50
K	5000	0.25	25	50
Na	5000	0.25	25	50
Ag	5	0.50	0.05	50
Ni	50	0.50	0.50	50
Sr	100	0.25	0.50	50
Ti	100	0.25	0.50	50

APPENDIX A

1.0 Application

Appendix A designed to supplement SOPs MET104.xx and MET105.xx for the preparation of soil samples for compliance with DoD and certain state-specific projects

2.0 Background

A theory of particulate sampling was developed by geologist Pierre Gy to improve the quality of data gathered in support of mineral exploration and mining. The MIS approach described herein is based upon Gy's theories and is applicable to environmental sampling at contaminated sites.

A large portion of sampling error is a result of compositional and distributional heterogeneity.

Compositional heterogeneity describes the variability of contaminant concentrations between the particles that make up the population in the sample. This type of heterogeneity results in fundamental error (FE).

Distributional heterogeneity occurs when particles are not randomly distributed across the population due to slight spatial variations. Spatial variability will be missed if all samples are collected from one place. This type of heterogeneity results in grouping and segregation error (GSE).

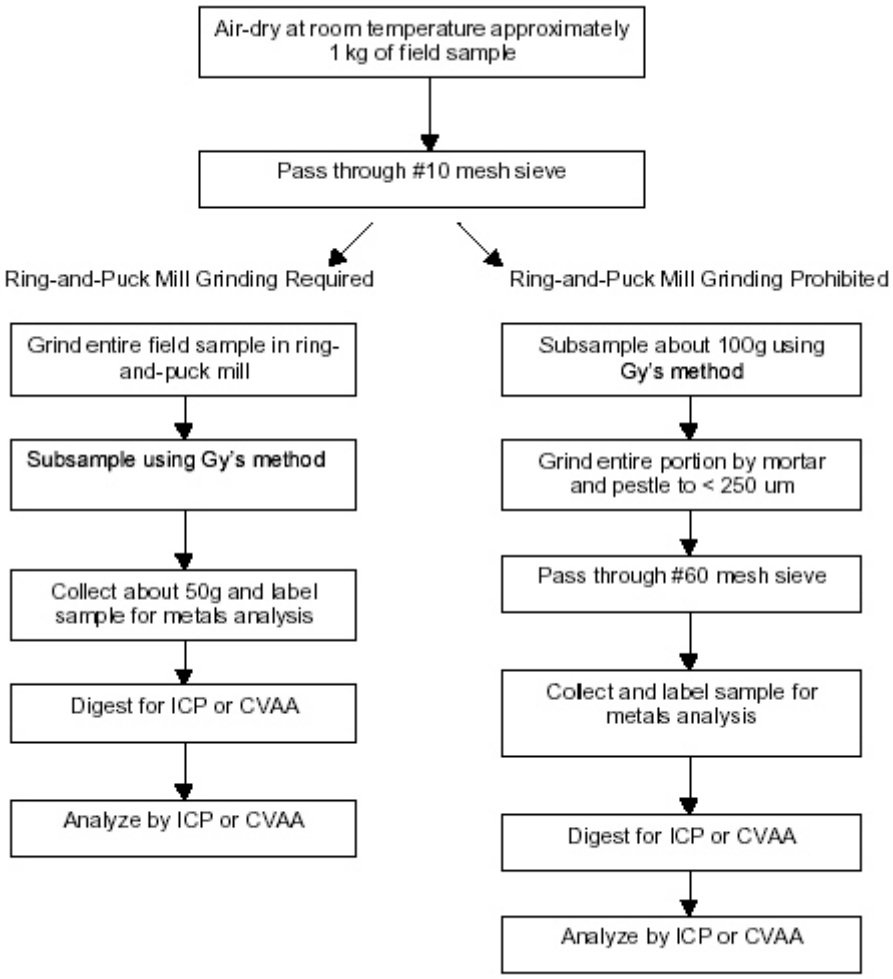
Gy found that fundamental error is directly proportionate to maximum particle size and inversely proportionate to sample size, therefore it is beneficial to collect and analyze a sample of sufficient size that consists of particulate matter where majority of contamination is present. In order to manage FE under 15%, particulate matter size must be under 2 mm and minimum sample mass above 30g.

To minimize GSE, it is imperative to collect sample increments randomly and in enough locations to capture the spatial variability, even within sample that already has been collected from the field.

3.0 Subsampling for Metals

Some projects require that metals analysis be performed on the multi-incremental sample that was collected for 8330B. The technique used should be listed in the project QAPP or SOW. Consult the client if this information is not available.

See flow chart below for various subsampling techniques:



If Ring and Puck Mill grinding is required, then proceed with the grinding procedure listed in SOP OP046 for explosives. The metallic components from the Ring and Puck Mill may introduce chromium and iron into the sample.

After grinding, place a baking tray on the downdraft table. Transfer the entire sample to the tray. Shape the sample into an elongated pile with flattened top surface that it is approximately 1 cm thick. Using a rectangular scoop, collect multiple top-to-bottom cuts across the sample (see figure below). A minimum of 4 cuts should be made through each sample. Combine the cuts in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Transfer the samples to the metals department for analysis.

If Ring and Puck Mill grinding is not required then follow the procedure listed below.

Transfer the sample to a large ziplock bag after it has been air dried and sieved. Sample should be transferred over the downdraft tables to minimize dust contamination. Seal the bag and thoroughly mix the sample.

Place a baking tray on the downdraft table. Transfer the entire sample to the tray. Shape the sample into an elongated pile with flattened top surface that it is approximately 1 cm thick. Using a rectangular scoop, collect multiple top-to-bottom cuts across the sample (see figure below). A minimum of 4 cuts should be made through each sample. Combine the cuts in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Return the remaining sample to the ziplock bag or mixing bowl.

Grind each sample and MB to a particle size less than 250 um with a non-metallic mortar and pestle.

Place a baking tray on the downdraft table. Sieve each sample through a #60 sieve onto a tray.

Collect and label the samples. Transfer the samples to the metals department for analysis.

For digestion withdraw approximately 5 g of sieved material. If mortar-and-pestle grinding was specified per QAPjP, 1 g is sufficient. Follow digestion procedure outlined in the body of this SOP.

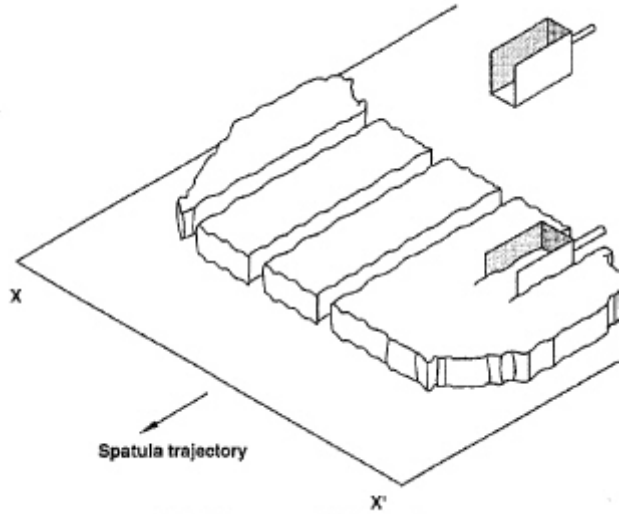


FIG. 1 Transversal Subsampling

ANALYSIS OF SEMIVOLATILE ORGANICS BY GC/MS SELECT ION MONITORING (SIM)

Prepared by: Norm Farmer Date: 05/14/14

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TEST NAME: ANALYSIS OF SEMIVOLATILE ORGANICS BY GC/MS SELECT ION MONITORING (SIM)

METHOD REFERENCE: SW846 8270D

DEPT: MS

Revised Sections: 1.1.6, 7.1, 7.4.1.1, 7.4.1.3, 7.4.1.4, 7.4.2.2, 7.4.2.4-7.4.2.6, 7.4.3.4, 7.4.3.5, 9.2.2.3, 9.4.1 and 9.4.2.4

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of various semivolatile organic compounds in water and solid matrices utilizing a gas chromatograph equipped with a mass spectrometer detector. Routine compounds can be found in Table 1.

1.1.2 Unlike convention full scan 8270; this method utilizes the instrument's select ion monitoring (SIM) capabilities. By monitoring for a few specific ions the sensitivity can be increased 10 to 20 fold.

1.1.3 Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. Reporting limits may vary depending on matrix complications and sample volumes. Reporting limits for this method are in the range of 0.2 to 1.0 ug/l for aqueous samples and 7 to 70 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.1.5 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

1.1.6 For DOD projects refer to QSM 4.2, Table F4 or QSM 5.0, Table 4 for additional method requirements and data qualifying guidance.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8270D.

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1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with Accutest SOP OP006, OP007, OP059 and OP060.

1.2.4 The extracts are analyzed on a gas chromatograph equipped with mass spectrometer detector.

1.2.5 The peaks detected are identified by comparison to characteristic ions and retention times specific to the known target list of compounds.

1.2.6 Library searches can not be performed on data acquired in SIM mode because data was only acquired for selected ions.

1.2.7 Manual integrations are performed in accordance with SOP QA029.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be refrigerated at –10°C to –20°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including chlorinated hydrocarbons, petroleum hydrocarbons, and phthalate esters may be coextracted by this method.

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3.4 SIM may provide a lesser degree of confidence in compound identification unless multiple ions are monitored for each compound. In general, Accutest monitors 3 ions per compound.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Internal Standards: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Internal standards for Mass Spec methods are often deuterated forms of target analytes. Internal standards are used to compensate for retention time and response shifts during an analytical run.

4.6 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

4.7 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

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4.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.12 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.13 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Methylene Chloride – pesticide grade or equivalent

5.2 Semivolatile stock standards – Various mixes, traceable to Certificate of Analysis

5.3 Decafluorotriphenylphosphine mix (DFTPP) – Also contains pentachlorophenol, benzidine and DDT.

5.4 Base/neutral surrogate standards – dependent on the analytes being analyzed

Nitrobenzene-d5
2-Fluorobiphenyl
P-Terphenyl-d14

5.5 Acid surrogate standards – dependent on the analytes being analyzed

Phenol-d6
2-Fluorophenol
2,4,6-Tribromophenol

5.6 Internal standards – dependent on the analytes being analyzed

1,4-Dichlorobenzene-d4	Naphthalene-d8
Acenaphthene-d10	Phenanthrene-d10
Chrysene-d12	Perylene-d12

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6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 with 7683 Autosampler

6.1.1 Gas Chromatograph

The analytical system that is complete with a temperature programmable gas chromatograph and all required accessories, analytical columns, and gases.

6.1.2 The injection port is designed for split-splitless injection with capillary columns.

6.1.3 Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Mass Spectrometer – Agilent Technologies 5973 and 5975

The mass spectrometer must be capable of scanning from 35-500 amu every second or less utilizing a 70-volt (nominal) electron energy in the electron impact ionization mode. It must also be capable of producing a mass spectrum that meets all the criteria in section 7.4.1.1 when injecting 50 ng of Decafluorotriphenylphosphine (DFTPP).

The mass spectrometer must be capable of analyzing multiple groups for up to 30 specific ions. The start and end of each group should be time programmable. Each group of specific ions is referred to as a descriptor.

6.3 Data System – Agilent Technologies MS Chemstation rev. DA 02.0x, DA 03.0x or EA 02.0x.

6.3.1 A computer system interfaced to the mass spectrometer that allows for the continuous acquisition and storage of all mass spectral data obtained throughout the duration of the chromatographic program.

6.3.2 The computer utilizes software that allows searching any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP).

6.3.3 The software should allow for integrating the abundances in any EICP between specific time or scan number limits. See Table 3.

6.3.4 Data is archived to magnetic tape for long term storage.

6.4 Column – TG-5MS or equivalent: 30m X 0.25mm X 0.25um
– Rxi-5SIL or equivalent: 30m X 0.25mm X 0.25um

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6.5 Gas-tight syringes and class “A” volumetric glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards should be stored at temperatures between –10 °C and –20 °C, or as recommended by the manufacturer. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the MS STD Summary in the Active SOP directory.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a “Certificate of Analysis” with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor’s expiration date. Once opened, the hold time is reduced to one year or the vendor’s expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with methylene chloride. The hold time for intermediate standards is six months or the vendor’s expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicates analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the semivolatile organics are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the RL and the remaining standards define the working range of the detector.

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions and Mass Spectrometer Descriptors

1ul or 2ul autosampler injection

Pulsed splitless or splitless

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Carrier gas – UHP Helium (7.7psi to 36psi @1.5 psi/min ramp pressure)

Injection port temperature – 280 °C Transfer line temperature – 280 °C

Source temperature – 230 °C Quad temperature – 150 °C

Oven program – 55 °C for 1.0 minutes
27.5 °C/min to 265 °C for 0 minutes
5 °C/min to 300 °C for 0 minutes
20 °C/min to 320 °C for 0 minutes

OR

Oven program – 40 °C for 1.5 minutes
30 °C/min to 190 °C for 0 minutes
10 °C/min to 260 °C for 0 minutes
25 °C/min to 320 °C for 2.0 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

MS Descriptors – Monitor 3 characteristic ions for each target analyte, and 2 characteristic ions for each surrogate and internal standard. Each descriptor may have up to 30 ions; however, the more ions in a descriptor, the less the sensitivity. Therefore, it is beneficial to use multiple descriptors for longer analytes lists.

GC conditions and mass spectrometer descriptors are dependent on the analytes being analyzed. Refer to the specific instrument methods for actual conditions and descriptors.

7.3 Sample Preparation

7.3.1 Water Samples

A 1000ml aliquot of sample is pH adjusted and extracted with methylene chloride utilizing separatory funnel extraction. The extract is concentrated to 1.0ml.

7.3.2 Solid Samples

A 30-gram aliquot of sample is extracted with methylene chloride and acetone utilizing pulse sonication or microwave extraction. The extract is concentrated to 1.0ml.

7.4 Gas Chromatographic Analysis

Instrument calibration consists of two major sections:

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Initial Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, the GC/MS system must be tuned, the injection port inertness must be verified, and the instrument must be calibrated.

7.4.1.1 Tune Verification (DFTPP)

The instrument should be hardware tuned per manufacturer's instructions. Verify the instrument tune by injecting 50ng of DFTPP solution onto the instrument. The resulting DFTPP spectra should meet the criteria in the following table.

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60 of mass 198
68	<2 % of mass 69
70	<2 % of mass 69
127	40-60 % of mass 198
197	<1 % of mass 198
198	Base peak, 100 % relative abundance
199	5-9 % of mass 198
275	10-30 % of mass 198
365	>1 % of mass 198
441	Present but less than mass 443
442	>40 % of mass 198
443	17-23 % of mass 442

Evaluate the tune spectrum using three mass scans from the chromatographic peak and a subtraction of instrument background. This procedure is performed automatically by the MS Chemstation software by running "autofind" on the DFTPP peak.

Select the scans at the peak apex and one to each side of the apex. Calculate an average of the mass abundances from the three scans.

Background subtraction is required. Select a single scan in the chromatogram that is absent of any interfering compound peak and no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tuning compound peak.

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If the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are met.

Analysis must not begin until the tuning criteria are met. The injection time of the acceptable tune analysis is considered the start of the 12-hour clock. The same mass spec settings must be used for the calibration standards and samples that were used for the tune evaluation standard. The exception being that the tune evaluation standard must be acquired in full scan mode and all others in SIM mode.

7.4.1.2 Injection Port Inertness Verification

DDT, pentachlorophenol, and benzidine must also be evaluated in the tune standard. These compounds are used to assess injection port inertness and column performance.

Pentachlorophenol and benzidine should be present at their normal responses and, no peak tailing should be visible. The tailing factor for Benzidine must be less than 2 and the tailing factor for pentachlorophenol must be less than 2.

DDT breakdown should not exceed 20%. Breakdown is calculated as follows:

$$\%DDT_{BREAKDOWN} = \frac{(\text{DDE Area} + \text{DDD Area}) \times 100}{(\text{DDE Area} + \text{DDD Area} + \text{DDT Area})}$$

If degradation is excessive or peak tailing is noticed, injection port maintenance is required.

This performance test must be passed before any samples or standards are analyzed.

7.4.1.3 Internal Standard Calibration

A minimum 5-point calibration curve is created for the semivolatile organic compounds and surrogates using an internal standard technique. Accutest Laboratories routinely performs a 6-point calibration to maximize the calibration range.

The low point may be omitted from the calibration table for any compound with an RL set at the level two standard. Additionally the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

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Response factors (RF) for each analyte are determined as follows:

$$RF = (A_{anayte} \mathbf{X} C_{istd})/(A_{istd} \mathbf{X} C_{analyte})$$

A_{anayte} = area of the analyte
A_{istd} = area of the internal standard
$C_{analyte}$ = concentration of the analyte
C_{istd} = concentration of the internal standard.

The mean RF and standard deviation of the RF are determined for each analyte. The percent relative standard deviation (%RSD) of the response factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of RF X 100) / Mean RF

If the %RSD ≤ 20%, linearity through the origin can be assumed and the mean RF can be used to quantitate target analytes in the samples. **The %RSD should be ≤ 15% for any DoD QSM projects.**

Alternatively if the %RSD > 20% a calibration curve of response vs. amount can be plotted. If the correlation coefficient (r) is ≥0.995 (r^2 ≥0.990) then the curve can be used to quantitate target analytes in the samples.

NOTE: If a linear regression is used for an analyte, then the low standard must be recalculated against the current initial calibration. The recovery of any analyte using a linear regression must be 70% -130% of the expected value. This requirement does not apply to quadratic regressions.

The method also employs minimum response factor (RF) criteria for select target analytes. See Table 2 for the analytes and associated minimum response factors. Unlike previous revisions that only set a minimum for the average RF, 8270D requires that the minimum RF be met for each level of the calibration curve.

7.4.1.4 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV should be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be ≤ 30%. If the %D > 30%, the analysis of samples may still proceed if the analyte

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failed high and the analyte is not expected to be present in the samples. However, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 30% in the ICV, the sample will need to be reanalyzed on a system with a passing ICV for that analyte.

For any DoD QSM project, the %D for all target compounds should be ≤ 20%. If samples must be analyzed with an analyte of interest having a %D > 20%, then the data must be qualified accordingly.

If the ICV does not meet this criteria, a second standard should be prepared. If the ICV still does not meet criteria, analyze an ICV prepared from a third source. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.4.2 Continuing Calibration Verification (CCV)

7.4.2.1 Inject 1ul of the tune evaluation mix at the beginning of each 12-hour shift. Evaluate the resultant peaks against the criteria in sections 7.4.1.1 and 7.4.1.2. The injection time of this standard starts the 12-hour window.

7.4.2.2. Analyze a continuing calibration check standard. The CCV should be at or below the mid-point of the calibration curve.

7.4.2.3. The response factor for any target analyte listed in Table 2 must meet the listed minimum value.

7.4.2.4. The %D for all other analytes of interest should be ≤ 20%. If the %D > 20%, the analysis of samples may still proceed if the analyte failed high and the analyte is not expected to be present in the samples. However, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 20% in the CCV, the sample will need to be reanalyzed on a system with a passing CCV for that analyte, or the data must be qualified.

For any DoD QSM project, the %D for all target compounds should be ≤ 20%. If samples must be reported with an analyte of interest having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

7.4.2.5. The criteria in 7.4.2.3 and 7.4.2.4 must be met for the continuing calibration to be considered valid. Only analytes that are being reported for a given sample must meet the criteria in 7.4.2.3 and

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7.4.2.4. If the first continuing calibration verification does not meet criteria, a second standard may be injected.

Rationale for second standard such as instrument maintenance, clipped column, remade standard, etc should be documented in the run log or maintenance log. Reanalysis of second standard without valid rationale may require the analysis of a third standard (in which case both the second and third standard would have to pass).

If the second standard (or third) does not meet criteria, the system must be recalibrated.

7.4.2.6. For QSM 5 compliance an additional CCV must be analyzed at the end of each run. The %D for all target compounds in this CCV should be ≤ 50%. If samples must be reported with an analyte of interest having a %D > 50%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

7.4.2.7. If any of the internal standard area change by a factor of two (-50% to +100%) or retention time changes by more than 30 seconds from the midpoint standard of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections made, as appropriate. Corrective action may include re-calibration (initial Calibration) of the instrument.

7.4.3 Sample Extract Analysis

7.4.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Tune Evaluation Mix
Initial Calibration Standards (or CCV)
QC Extracts
Sample Extracts

7.4.3.2 Two microliters of internal standard solution is added to every 100ul of extract in the autosampler vial. Generally, 400ul of extract are transferred to the autosampler vial with a gas tight syringe.

7.4.3.3 One or two microliters (same amount as standards) of extract is injected into the GC by the autosampler. The data system then records the resultant peak responses and retention times.

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7.4.3.4 Qualitative identification

The target compounds shall be identified by analysts with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. The criteria required for a positive identification is:

The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

The sample component must elute at the same relative retention time (RRT) as the daily standard. The RRT of sample component must be within ± 0.06 RRT units of the standard.

All ions monitored in the standard mass spectra should be present in the sample spectrum.

The relative intensities of these ions must agree within ± 30% between the daily standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%.

Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

If peak identification is prevented by the presence of interferences, further cleanup may be required or the extract must be diluted so that the interference does not mask any analytes.

7.4.3.5 Quantitative analysis

When a target compound has been identified, concentration will be based on the integrated area of the quantitation ion, which is normally the base peak.

The sample matrix may produce an interference with the primary ion. This may be characterized by an excessive background

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signal of the same ion, which distorts the peak shape beyond a definitive integration. The interference could also, severely inhibit the response of the internal standard ion.

If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.5 Maintenance and Trouble Shooting

7.5.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.5.2 All instrument maintenance must be documented in the appropriate "Instrument Repair and Maintenance" log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.5.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.5.4 PC and software changes must be documented in the "Instrument Repair and Maintenance" log. Software changes may require additional validation.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

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9.1 Internal Standards

9.1.1 1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12 and Perylene-d12 can be used as internal standards for this method. The internal standards used are dependent on the compounds being analyzed. The response of the internal standard in all subsequent runs should be within a factor of two (-50% to +100%) of the internal standard response in the opening CCV for each sequence. On days that an initial calibration is performed, the internal standard responses should be compared to the internal standard responses for the mid-point standard.

9.1.2 If the internal standard responses are not within limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, integrations, or internal standards solutions. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, prepare a second aliquot of extract and reanalyze the sample.

9.1.2.4 If upon reanalysis, the responses are still not within limits, the problem is considered matrix interference. The extract may need to be diluted or the results qualified.

9.2 Surrogates

9.2.1 Nitrobenzene-d5, 2-Fluorobiphenyl, and p-Terphenyl-d14 are used as the base neutral surrogate standards and Phenol-d5, 2-Fluorophenol, and 2,4,6-Tribromophenol are used as the acid surrogate standards to monitor the efficiency of the extraction. The surrogates used are dependent on the compounds being analyzed.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for all surrogates for the results to be acceptable.

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9.2.2 If any surrogate recovery is not within the established control limits, the following are required. Note: If the samples are being analyzed for only base neutral compounds or only acid compounds, then only the relative surrogates need to be monitored.

9.2.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, surrogate solutions or internal standard solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.2.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.2.2.3 If no problem is found, re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary. For any DoD QSM projects the resulting data must be qualified accordingly. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.2.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

9.3 Method Blank

9.3.1 The method blank is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high level samples. The method blank must be free of any analytes of interest or interferences at ½ the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.3.2 If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

9.3.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be

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investigated and documented. The samples results may be reported with the appropriate "B" or "V" qualifier. This must be approved by the department supervisor.

9.3.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination should be investigated and documented. The samples should be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4 Blank Spike

9.4.1 The blank spike is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

Marginal exceedances are not permitted for analytes that are deemed to be "Compounds of Concern" for a specific project. "Compounds of Concern" are different from "Target Compounds". "Target Compounds" are all analytes that are being reported for a site where "Compounds of Concern" are those analytes expected to be present at the site.

The number of allowable marginal exceedances is as follows:

1) 31-50 analytes in BS, 2 analytes allowed in ME range;

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2) 11-30 analytes in BS, 1 analyte allowed in ME range;

3) < 11 analytes in BS, no analytes allowed in ME range

9.4.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standard solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.4.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.4.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary and the sample results can be reported without qualification.

9.4.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable. For any DoD QSM projects the resulting data must be qualified accordingly.

9.4.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.4.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4.2.7 Because of their problematic nature, benzidine, benzaldehyde, and benzoic acid are generally not evaluated in the blank spike unless they are of specific concern for a given project.

9.5 Matrix Spike and Matrix Spike Duplicate

9.5.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all

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cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.5.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.5.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standard solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.5.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.5.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extraction but are an indication of the sample matrix effects.

9.5.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

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10.0 CALCULATIONS

The concentration of each analyte in the original sample is calculated as follows:

Water (ug/l) = $(CONC_{inst}) \times (V_F / V_I) \times DF$

Soil (ug/kg) = $[(CONC_{inst}) \times (V_F / W_I) \times DF] / \%solids$

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean RF, linear curve, or quadratic curve
DF	=	Dilution Factor
V_F	=	Volume of final extract (ul)
V_I	=	Volume of sample extracted (ml)
W_I	=	Weight of sample extracted (g)
%solids	=	Dry weight determination in decimal form

All soils are reported on a dry weight basis.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses, gloves, and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

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12.0 REFERENCES

SW846 Method 8000C Revision 3, March 2003

SW846 Method 8270D Revision 4, February 2007

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TABLE 1

Routine Target Analytes

Naphthalene
2-Methylnaphthalene
1-Methylnaphthalene
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene
Benzo[a]anthracene
Chrysene
Benzo[b]fluoranthene
Benzo[k]fluoranthene
Benzo[a]pyrene
Indeno[1,2,3-cd]pyrene
Dibenz[a,h]anthracene
Benzo[g,h,i]perylene
Carbazole
Pentachlorophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Hexachlorobenzene
Hexachlorobutadiene
bis(2-Chloroethyl)ether
2,4-Dichlorophenol
1,4-Dioxane
1,1'-Biphenyl
Diphenyl Ether
4-Nitrophenol
2,4-Dinitrophenol
N-Nitroso-di-n-propylamine
N-nitrosodimethylamine
Nitrobenzene
Dibenzofuran
bis(2-Ethylhexyl)phthalate
Hexachlorocyclopentadiene

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TABLE 2

Minimum Response Factors

Analyte	Min. RF	Analyte	Min. RF
Naphthalene	0.700	N-nitroso-di-n-propylamine	0.500
2-Methylnaphthalene	0.400	Hexachlorocyclopentadiene	0.050
1-Methylnaphthalene	0.400	2,4-Dinitrophenol	0.010
Acenaphthylene	0.900	4-Nitrophenol	0.010
Acenaphthene	0.900	Pentachlorophenol	0.050
Fluorene	0.900	bis(2-chloroethyl)ether	0.700
Phenanthrene	0.700	2,4-Dinitrotoluene	0.200
Anthracene	0.700	2,6-Dinitrotoluene	0.200
Fluoranthene	0.600	Hexachlorobenzene	0.100
Pyrene	0.600	1,4-Dioxane	n/a
Benzo[a]anthracene	0.800	1,1' Biphenyl	0.010
Chrysene	0.700	Diphenyl Ether	n/a
Benzo[b]fluoranthene	0.700	Carbazole	0.010
Benzo[k]fluoranthene	0.700	Dibenzofuran	0.800
Benzo[a]pyrene	0.700	bis(2-Ethylhexyl)phthalate	0.010
Indeno[1,2,3-cd]pyrene	0.500		
Dibenz[a,h]anthracene	0.400		
Benzo[g,h,i]perylene	0.500		

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TABLE 3

Characteristic Ions

Analyte	Quant. Ion	Q1	Q2
Naphthalene-d8 IS	136	68	
Nitrobenzene-d5 SS	82	128	54
Naphthalene	128	129	127
2-Methylnaphthalene	142	141	115
1-Methylnaphthalene	142	141	115
Acenaphthene-d10 IS	164	162	160
2-Fluorobiphenyl SS	172	171	
Acenaphthylene	152	151	153
Acenaphthene	153	152	154
Fluorene	166	165	167
Phenanthrene-d10 IS	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Fluoranthene	202	101	203
Chrysene-d12 IS	240	120	236
Pyrene	202	101	203
Terphenyl-d14 SS	244	122	212
Benzo[a]anthracene	228	226	229
Chrysene	228	226	229
Perylene-d12 IS	264	260	265
Benzo[b]fluoranthene	252	253	125
Benzo[k]fluoranthene	252	253	125
Benzo[a]pyrene	252	253	125
Indeno[1,2,3-cd]pyrene	276	138	277
Dibenz[a,h]anthracene	278	139	279
Benzo[g,h,i]perylene	276	138	277

Analyte	Quant. Ion	Q1	Q2
N-nitroso-di-n-propylamine	42	70	
Hexachlorocyclopentadiene	237	235	
2,4-Dinitrophenol	184	154	
4-Nitrophenol	139	65	
2,4,6-Tribromophenol	330	332	141
Pentachlorophenol	266	264	268
bis(2-chloroethyl)ether	93	63	95
2,4-Dinitrotoluene	165	63	182
2,6-Dinitrotoluene	165	89	121
Hexachlorobenzene	284	142	249
1,4 Dioxane	88	58	43
1,1' Biphenyl	154	153	152
Diphenyl Ether	170	141	77
Carbazole	167	166	139
Dibenzofuran	168	139	169
bis(2-Ethylhexyl)phthalate	149	167	279

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STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF BASE-NEUTRAL AND ACID (BNAs) EXTRACTABLES FROM WATER SAMPLES

Prepared by:	Norm Farmer	Date:	08/26/13
Reviewed by:	Mark Erstling	Date:	09/04/13

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TEST NAME: **STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF BASE-NEUTRAL AND ACID (BNAs) EXTRACTABLES FROM WATER SAMPLES**

Method: EPA 625 and SW846 3510C/8270D

Dept: OP

Revised Sections: 3.2.1, 7.1, 7.2, 7.3, 7.8-7.18 and 9.1.2

1.0 Summary, Scope and Application

1.1 Summary

Aqueous samples are serially extracted with methylene chloride, concentrated by Kuderna-Danish apparatus, and stored in glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to aqueous samples submitted for semivolatile analysis by GC/MS methods, including EPA 625 and SW-846 8270D.

2.0 Discussion and Comments

This procedure is adapted from SW-846 methods 3500C and 3510C. The method utilizes “Separatory Funnel Liquid-Liquid Extraction”; it is not applicable to samples requiring “Continuous Liquid-Liquid Extraction”.

Although the mass spectrometer identifies compounds by specific ions, it will respond to most organic compounds. It is important to minimize extraneous contaminants by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass or Teflon. Plastic items must be avoided because they can lead to phthalate contamination.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples.

3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be refrigerated at –10°C to –20°C until analysis.

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3.2 Holding Time

3.2.1 Aqueous samples must be extracted within 7 days of collection. The Date/Time that the extraction is started and completed must be recorded on the prep sheet.

3.2.2 Extracts must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

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4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetone – pesticide grade or equivalent

5.2 Methylene chloride – pesticide grade or equivalent

5.3 Anhydrous sodium sulfate – precleaned to remove phthalates

5.4 Reagent water – distilled or deionized - free of interferences

5.5 10 Normal NaOH

5.6 1:1 H_2SO_4

5.7 BNA Surrogate Solution – prepared in acetone or methanol at a concentration specified by the GC/MS analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.8 BNA Spike Solution #1– prepared in acetone or methanol at a concentration specified by the GC/MS analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use. This solution contains the majority of the spiked analytes.

5.9 BNA Spike Solution #2– prepared in acetone or methanol at a concentration specified by the GC/MS analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use. This solution contains analytes that are not stable when mixed with those in Solution #1.

6.0 Glassware and Apparatus

6.1 1000ml graduated cylinder

6.2 2 liter separatory funnel (glass or Teflon)

6.3 250ml separatory funnel

6.4 250ml or 500ml Erlenmeyer flasks

6.5 0.5ml or 1.0ml syringes

6.6 1.0ml volumetric flask

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6.7 500ml Kuderna-Danish (K-D) flask

6.8 10ml graduated concentrator tube

6.9 3-ball macro Snyder column

6.10 Disposable transfer pipettes

6.11 pH paper

6.12 Teflon boiling chips

6.13 glass wool – precleaned

6.14 Glass filter funnel

6.15 Fisher P8 filters, or equivalent

6.16 2.0ml glass screw cap vials – caps must have Teflon lined septa

6.17 Water bath – adjustable temperature control

6.18 Nitrogen evaporator

7.0 Procedure

7.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: batch number, sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, extraction dates and times, and extraction technician.

The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC/MS analyst with the extracts. The Batch number, extraction technician, and extraction start Date and Time are entered into LIMS.

7.2 Sample Transfer

7.2.1 Mark the level of the sample (upper edge) on the bottle with a marker. Transfer the entire sample directly into the appropriately labeled separatory funnel. Add 50-60ml methylene chloride to the sample bottle. Cap the bottle and invert several times to thoroughly rinse the walls and cap. Transfer the solvent to the appropriate separatory funnel.

CAUTION: ALL SOLVENT ADDITIONS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

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7.2.2 Fill the bottle to the sample mark with tap water. Transfer the water to a 1000ml graduated cylinder and record the sample volume. Discard the tap water.

7.2.3 **Alternative procedure for samples with high solids content.**

The entire contents of the sample bottle should be analyzed for aqueous samples, including any solids that may have been collected. However, high levels of solids in the sample can create heavy emulsions that can not be broken down by the various mechanical means.

The solids will normally settle out during storage. If the sample bottle contains more than an inch of solids, it may be necessary to decant the water phase rather than extracting the entire sample. The decision to decant a water sample should be based on the experience and judgement of the extraction technician or the department supervisor. If the sample is decanted, it must be noted on the prep sheet.

Using a 1000ml-graduated cylinder, transfer the sample to the appropriately labeled separatory funnel taking care to minimize the amount of solids that are transferred. Record the actual sample volume that is transferred. Rinse the graduated cylinder with a 50-60ml aliquot of methylene chloride and transfer it to the appropriate separatory funnel.

CAUTION: ALL SOLVENT ADDITIONS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

The graduated cylinder must be rinsed with tap water, reagent water, and methylene chloride between samples in order to prevent cross contamination.

7.3 Using a 1000ml-graduated cylinder, transfer each of the QC samples to the appropriately labeled separatory funnels. This includes the method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD). Use 1000ml of reagent water for the MB and BS. Use additional sample aliquots for the MS and MSD. If there is insufficient sample volume, a lesser volume may be used. Record both the sample ID and volume on the prep sheet.

7.3.1 Rinse the graduated cylinder with a 50-60ml aliquot of methylene chloride and transfer it to the appropriate separatory funnel. Transfer the solvent to the appropriate separatory funnel.

7.3.2 If entire bottles were used for MS and MSD, add 50-60ml methylene chloride to each bottle. Cap the bottle and invert several times to thoroughly rinse the walls and cap. Transfer the solvent to the appropriate separatory funnel.

7.4 Check the pH of each sample by dipping a disposable transfer pipette into the sample and touching it to the pH paper. Record the pH on the prep sheet.

7.5 Using the dedicated surrogate syringe add 0.5ml of surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

7.6 Using the dedicated spike syringe add 0.5ml of spike solution #1 and #2 to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

NOTE: If samples are being analyzed for Base-Neutral extractable compounds only, sections 7.7 to 7.10 may be omitted.

7.7 Adjust the pH of each sample to <2 by adding 1ml aliquots of 1:1 H_2SO_4. Swirl the sample and recheck the pH after each aliquot is added.

7.8 Cap and shake each separatory funnel for two minutes.

CAUTION: THE SEPARATORY FUNNELS MUST BE PERIODICALLY VENTED TO AVOID AN EXCESSIVE BUILDUP IN PRESSURE. THIS SHOULD BE DONE IN A HOOD.

7.9 After shaking, allow the layers to separate for at least 10 minutes. Collect the solvent layer (bottom) in a labeled 500ml Erlenmeyer flask.

NOTE: Some samples may form emulsions. If emulsions are present, the technician must take steps to breakdown the emulsion. This may include filtering the emulsion through a smaller separatory funnel, centrifuging, or filtering through sodium sulfate.

7.10 Repeat steps 7.8 and 7.9 two additional times using 50 to 60ml of methylene chloride. Combine the extract in the Erlenmeyer flask.

NOTE: If samples are being analyzed for Acid extractable compounds only, sections 7.11 to 7.15 may be omitted.

7.11 Adjust the pH of each sample to >11 by adding 2ml aliquots of 10 N NaOH. Swirl the sample and recheck the pH after each aliquot is added.

7.12 Add 50 to 60ml methylene chloride to each sample. If samples are being extracted for Base-Neutral extractable compounds only, then the separatory funnel will already contain 50 to 60 ml of methylene chloride.

7.13 Cap and shake each separatory funnel for two minutes.

CAUTION: THE SEPARATORY FUNNELS MUST BE PERIODICALLY VENTED TO AVOID AN EXCESSIVE BUILDUP IN PRESSURE. THIS SHOULD BE DONE IN A HOOD.

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7.14 After shaking, allow the layers to separate for at least 10 minutes. Collect the solvent layer (bottom) in a labeled 500ml Erlenmeyer flask. Acid and Base fractions may be combined in the same Erlenmeyer flask.

NOTE: Some samples may form emulsions. If emulsions are present, the technician must take steps to breakdown the emulsion. This may include filtering the emulsion through a smaller separatory funnel, centrifuging, or filtering through sodium sulfate.

7.15 Repeat steps 7.13 and 7.14 two additional times using 50 to 60ml of methylene chloride. Combine the extract in the Erlenmeyer flask.

7.16 If the entire extraction procedure can not be completed on the same day, the Erlenmeyer flasks may be covered with aluminum foil and refrigerated.

7.17 Assemble and label the K-D flasks and concentrator tubes. Using forceps, place a Teflon boiling chip in each concentrator tube.

7.18 This step is **mandatory** for this extraction because any acid residue remaining in the extract will cause some of the unstable analytes to decompose. Place a glass filter funnel containing sodium sulfate supported on a Fisher P8 filter on the K-D flask. Pour the extracts through the sodium sulfate into the K-D flasks and rinse with methylene chloride. Remove the funnels and attach the Snyder columns.

CAUTION: ALL EXTRACT TRANSFERS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

7.19 Pre-wet the Snyder column with a few drops of methylene chloride and place the K-D assembly in a hot (65 to 75 ^{o}C) water bath. **NOTE:** If the bath is too hot, the more volatile compounds may be lost during this step. Concentrate the extract to approximately 5ml.

7.20 Remove the K-D assembly from the bath and allow it to cool. Remove the Snyder columns and rinse them with methylene chloride and then with acetone before placing them back in their storage rack.

7.21 Wipe the water from the joint area between the K-D flask and the concentrator tube. Transfer the sample ID to the concentrator tube. Remove the concentrator tube from the K-D flask, **rinse the joint with solvent**, and place the concentrator tube in the rack for the nitrogen evaporator.

7.22 Use a steady stream of nitrogen to concentrate the extract to 0.5ml.

7.23 If the extract is cloudy or contains water droplets, run the extract through a micro column of glass wool and sodium sulfate.

7.24 Transfer the extract to a 1.0ml volumetric flask. Rinse the concentrator tube with a few drops of methylene chloride and transfer it to the volumetric flask. Adjust

the final volume to 1.0ml. **NOTE:** If the extract will not concentrate to 1.0ml, choose the next appropriate final volume. Be sure to record the final volume on the prep sheet.

7.25 Transfer the extract to an appropriately labeled 2.0ml screw cap vial. Store the extracts in the "extract freezer" until they are needed for analysis.

8.0 Quality Assurance and Quality Control

8.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

8.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

9.0 Safety and Waste Disposal

9.1 Safety

9.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

9.1.2 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

9.1.3 Methylene chloride is an inhalation hazard and a suspected carcinogen. Fume hoods must be used to minimize exposure to vapors.

9.2 Waste Disposal

9.2.1 Waste methylene chloride is placed in the "chlorinated waste" container.

9.2.2 Waste acetone is placed in the "non-chlorinated waste" container.

9.2.3 Waste sodium sulfate is placed in a waste container after the solvent has drained or evaporated.

9.2.4 Extracted water samples are rinsed down the drain with large amounts of water.

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9.2.5 Samples are archived and stored for 30 days after analysis. After the storage time has elapsed, the remaining aqueous samples are transferred to the appropriate drums for disposal.

10.0 References

SW-846 Method 3500C, Rev. 3, 02/07

SW-846 Method 3510C, Rev. 3, 12/96

SW-846 Method 8270D, Rev. 4, 02/07

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STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF BASE-NEUTRAL AND ACID (BNAs) EXTRACTABLES FROM SOLID SAMPLES

Prepared by:	Norm Farmer	Date:	08/24/13
Reviewed by:	Mark Erstling	Date:	09/04/13

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TEST NAME: **STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF BASE-NEUTRAL AND ACID (BNAs) EXTRACTABLES FROM SOLID SAMPLES**

Method: SW846 3550C/8270D

Dept: OP

Revised Sections: 3.2.1, 7.0, 8.1, 8.6-8.18 and 10.1.4

1.0 Summary, Scope and Application

1.1 Summary

Solid samples are serially extracted by pulse sonication, concentrated by Kuderna-Danish apparatus, and stored in glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to solid samples, including soils and sediments, submitted for BNA analysis by GC/MS method SW-846 8270D.

2.0 Discussion and Comments

This procedure is adapted from SW-846 methods 3500C and 3550C. The method outlined in this SOP is designed for low concentration samples (concentration of the individual organic components is expected to be less than 20mg/kg).

Although the mass spectrometer identifies compounds by specific ions, it will respond to most organic compounds. It is important to minimize extraneous contaminants by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass, stainless steel, wood, or Teflon. Plastic items must be avoided because they can lead to phthalate contamination.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.1 Samples shall be collected in glass jars with Teflon lined caps. 250ml jars are recommended for solid samples.

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3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be refrigerated at –10°C to –20°C until analysis.

3.2 Holding Time

3.2.1 Solid samples must be extracted within 14 days of collection. The Date/Time that the extraction is started and completed must be recorded on the prep sheet.

3.2.2 Extracts must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

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4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetone – pesticide grade or equivalent

5.2 Methylene chloride – pesticide grade or equivalent

5.3 Anhydrous sodium sulfate – precleaned to remove phthalates

5.4 BNA Surrogate Solution – prepared in acetone or methanol at a concentration specified by the GC/MS analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.5 BNA Spike Solution #1– prepared in acetone or methanol at a concentration specified by the GC/MS analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use. This solution contains the majority of the spiked analytes.

5.6 BNA Spike Solution #2– prepared in acetone or methanol at a concentration specified by the GC/MS analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use. This solution contains analytes that are not stable when mixed with those in Solution #1.

6.0 Glassware and Apparatus

6.1 400ml thick walled beaker

6.2 Glass funnels (large enough to support the filters)

6.3 500ml Erlenmeyer flask

6.4 Spatula – stainless steel, wood, or Teflon

6.5 0.5ml or 1.0ml syringes

6.6 1.0ml volumetric flask

6.7 500ml Kuderna-Danish (K-D) flask

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6.8 10ml graduated concentrator tube

6.9 3-ball macro Snyder column

6.10 Disposable transfer pipettes

6.11 Teflon boiling chips

6.12 Glass wool – precleaned

6.13 Fisher P8 filters – or equivalent

6.14 2.0ml glass screw cap vials – caps must have Teflon lined septa

6.15 Water bath – adjustable temperature control

6.16 Nitrogen evaporator

6.17 Ultrasonic disrupter – minimum power of 300 watts with pulse capability

6.18 Disrupter horns – ¾" solid titanium tip

6.19 Top loading balance – capable of weighing samples to +/- 0.1 grams

7.0 Sonic Disrupter

The Misonix S3000 and Qsonica Q500 dual horn sonic disrupters do not require tuning. The sonic disrupters should be set in the pulse mode with a 50% duty cycle (energy on 50% of the time and off 50% of the time). The Misonix S3000 dual horn sonic disrupters will adjust the amplitude automatically to deliver 300 watts to the disrupter horns. The Qsonica Q500 sonic disrupters have a maximum power output of 500 watts. The power level should be set at 60-65% to deliver a minimum of 300 watts to the disrupter horns. Higher power settings may be used.

Read and follow the manufacturer's instructions for operating the sonic disrupters. Manufacturer's instructions may be found in the Active SOP directory. Any instrument maintenance or repairs in should be documented in the "Instrument Repair and Maintenance" logbook.

8.0 Procedure

8.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: batch number, sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, extraction dates and times, and extraction technician.

The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC/MS analyst with the extracts. The Batch number, extraction technician, and extraction start Date and Time are entered into LIMS.

8.2 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. Thoroughly mix the sample with a wooden spatula. Samples that are tightly packed or contain obvious layers may need to be transferred to a larger container for proper mixing. Refer to SOP QA034 for more information on sample homogenization.

8.3 Transfer approximately 30 grams of each sample to the appropriately labeled beakers. Use a clean spatula for each sample. Record the weight to the nearest 0.1gram on the prep sheet.

8.4 Add approximately 30 grams of sodium sulfate to each sample and mix until each sample has a free flowing sandy texture. Wetter soils will require more sodium sulfate.

8.5 Transfer approximately 30 grams of each of the QC samples to the appropriately labeled beakers. This includes the method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD). Use 30 grams of sodium sulfate and/or clean sand for the MB and BS. Use additional 30 gram aliquots of a sample for the MS and MSD. If there is insufficient sample amount, a lesser amount may be used. Record the sample ID, bottle number and weight on the prep sheet.

8.6 Using the dedicated spike syringe add 0.5ml of spike solution #1 and #2 to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

8.7 Using the dedicated surrogate syringe add 0.5ml of surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

8.8 Immediately add 100ml of 80:20 methylene chloride and acetone to each of the beakers. This will minimize the loss of the more volatile analytes.

NOTE: Crushed concrete samples should be extracted with 100ml aliquots of methylene chloride. Concrete reacts with the acetone to form excessive Aldol Condensation by-products that interfere with the early eluting acid target compounds and surrogates.

CAUTION: ALL SOLVENT ADDITIONS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

8.9 Label the Erlenmeyer flasks and place a glass filter funnel containing a Fisher P8 filter on the top of each flask.

8.10 Place a glass filter funnel containing a Fisher P8 filter on the top of each Erlenmeyer flask. **NOTE:** Samples may also be filtered directly into the K-D's.

8.11 Place the disrupter horn in the beaker such that the tip of the horn is approximately ½ inch into to solvent but not touching the beaker or the sample.

8.12 Sonicate the sample for 3 minutes.

8.13 Remove the beaker and decant the solvent through the filter funnel into the Erlenmeyer flask.

8.14 Repeat steps 8.11 to 8.13 two more times with additional 100ml solvent aliquots. All three solvent aliquots will be combined in the K-D flask.

8.15 After the final extraction, transfer the entire sample to the funnel, rinse thoroughly, and allow the solvent to drain through the sample.

NOTE: It is important to clean the disrupter horn between samples. This is done by rinsing the horn with the extraction solvent and wiping it dry with a Kim-wipe or paper towel.

8.16 If the entire extraction procedure can not be completed on the same day, the Erlenmeyer flasks may be covered with aluminum foil and refrigerated.

8.17 Assemble and label the K-D flasks and concentrator tubes. Using forceps, place a Teflon boiling chip in each concentrator tube and set each K-D flask in a support stand.

8.18 Transfer each extract to the appropriately labeled K-D. Rinse each Erlenmeyer flask with methylene chloride and transfer it to the appropriate K-D. Attach a Snyder column to each K-D flask.

CAUTION: ALL EXTRACT TRANSFERS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

8.19 Pre-wet the Snyder column with a few drops of methylene chloride and place the K-D assembly in a hot (75 to 85 ^{o}C) water bath. **NOTE:** If the bath is too hot, the more volatile compounds may be lost during this step. Concentrate the extract to approximately 5ml.

8.20 Remove the K-D assembly from the bath and allow it to cool. Remove the Snyder columns and rinse them with methylene chloride and then with acetone before placing them back in their storage rack.

8.21 Wipe the water from the joint area between the K-D flask and the concentrator tube. Be sure that the sample ID is still on the concentrator tube. Remove the concentrator tube from the K-D flask, **rinse the joint with solvent**, and place the concentrator tube in the rack for the nitrogen evaporator.

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8.22 Use a steady stream of nitrogen to concentrate the extract to 0.5ml.

8.23 If the extract is cloudy or contains water droplets, run the extract through a micro column of glass wool and sodium sulfate.

8.24 Transfer the extract to a 1.0ml volumetric flask. Rinse the concentrator tube with a few drops of methylene chloride and transfer it to the volumetric flask. Adjust the final volume to 1.0ml. **NOTE:** If the extract will not concentrate to 1.0ml, choose the next appropriate final volume. Be sure to record the final volume on the prep sheet.

8.25 Transfer the extract to an appropriately labeled 2.0ml screw cap vial. Store the extracts in the "extract freezer" until they are needed for analysis.

9.0 Quality Assurance and Quality Control

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

9.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

10.0 Safety and Waste Disposal

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

10.1.2 **Hearing protection** must be worn while operating the sonic disrupters. The high frequency could cause permanent hearing loss.

10.1.3 Avoid touching the disrupter horns while they are active. Contact may cause burns or tissue damage.

10.1.4 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

10.1.5 Methylene chloride is an inhalation hazard and a suspected carcinogen. Fume hoods must be used to minimize exposure to vapors.

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10.2 Waste Disposal

10.2.1 Waste methylene chloride is placed in the “chlorinated waste” container.

10.2.2 Waste acetone is placed in the “non-chlorinated waste” container.

10.2.3 Extracted soil samples are placed in a waste container after the solvent has drained or evaporated.

10.2.4 Waste soil from the homogenizing process should be placed in the “soil waste” container. **NOTE:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

10.2.5 Samples are archived and stored for 30 days after analysis. After the storage time has elapsed, the remaining soil samples are transferred to the appropriate drums for disposal.

11.0 References

SW-846 Method 3500C, Rev. 3, 02/07

SW-846 Method 3550C, Rev. 3, 02/07

SW-846 Method 8270D, Rev. 4, 02/07

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STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF SEMIVOLATILE ORGANICS AND METALS (TCLP)

Prepared by: Norm Farmer Date: 08/08/14

Approved by: Mark Erstling Date: 08/15/14

Annual Review

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TEST NAME: STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF SEMIVOLATILE ORGANICS AND METALS (TCLP)

Method: SW846 1311

Dept: OP

Revised Sections: 5.7, 7.2.8, 7.4.1, 7.4.2 and 7.5.4

1.0 Summary, Scope and Application

1.1 Summary

For liquid and aqueous samples containing less than 0.5% solids, the sample is filtered through TCLP filter paper and the filtrate is defined as the TCLP leachate. The leachate can then be analyzed for semivolatile organics and metals.

For solid samples, the solid portion of the sample is extracted by adding extraction fluid equal to 20 times the weight of the sample and rotating the sample for 18 hours at 30 rpm. The extraction fluid used is based on the alkalinity of the solid portion of the sample. After leaching, the sample is filtered through TCLP filter paper. The leachate can then be analyzed for semivolatile organics and metals.

1.2 Scope and Application

This procedure is applicable to samples submitted for TCLP semivolatile analysis and/or TCLP metals analysis.

1.2.1 Metals by 6010

1.2.2 Mercury by 7470

1.2.3 Semivolatiles by 8270

1.2.4 Pesticides by 8081

1.2.5 Herbicides by 8151

1.2.6 Extractable TPH by 8015

2.0 Discussion and Comments

This procedure is adapted from SW-846 method 1311. The method utilizes an extraction bottle and rotary agitation device to evaluate the presence and mobility of semivolatile analytes and metals. It is not applicable for evaluating the mobility of volatile analytes.

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3.0 Preservation and Holding Times

3.1 Preservation

3.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples. Liquid samples for the analysis of metals only may be collected in HDPE bottles.

3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until leaching.

3.1.3 Samples for TCLP analysis should not be chemically preserved prior to leaching. After filtration, the TCLP leachate for metals should be preserved to a pH <2 with nitric acid unless the leachates will be digested immediately.

3.1.4 TCLP Leachates for semivolatile organics must be protected from light and stored at ≤ 6°C from the time of filtration until extraction.

3.2 Holding Time

3.2.1 Samples submitted for the analysis of semivolatile organics including pesticides and herbicides must be leached within 14 days of collection.

3.2.2 Samples submitted for the analysis of mercury must be leached within 28 days of collection.

3.2.3 Samples submitted for the analysis of metals (except mercury) must be leached within 180 days of collection.

3.2.4 Leachates for semivolatile organics must be extracted by the appropriate procedure within 7 days of filtration.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

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4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Leachate Blank Spike (LBS): An aliquot of TCLP fluid spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Leachate blank spike recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.8 Leachate Blank (LB): An aliquot of TCLP fluid to which all reagents are added in the same volumes or proportions as used in sample processing. The leachate blank is processed simultaneously with the samples through all the steps of the analytical procedure. The Leachate blank is used to document contamination resulting from the analytical process.

4.9 Leachate Spike (LS): A sample leachate aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The leachate spike recoveries are used to document the bias of a method in a given sample matrix.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

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5.0 Reagents

5.1 Reagent water – distilled or deionized - free of interferences

5.2 Hydrochloric Acid, 1.0N Fisher brand or equivalent

5.3 Sodium Hydroxide, 10.0N Fisher brand or equivalent

5.4 Glacial Acetic Acid, Reagent Grade

5.5 Extraction Fluid #1: Add 57 ml of glacial acetic acid and 64.3 ml of 10.0N sodium hydroxide to a 1000ml graduated cylinder and dilute to 1 liter with reagent water. Transfer to a PTFE lined carboy. Using a 1000ml graduated cylinder, transfer an additional 9 liters of reagent water to the carboy. Mix thoroughly. This will prepare 10 liters of Fluid #1. The preparation of all TCLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use. The pH of this solution should be 4.93 $\pm$ 0.05.

NOTE: If the pH of the fluid is out of range, remake the fluid.

5.6 Extraction Fluid #2: Add 57 ml of glacial acetic acid to a 1000ml graduated cylinder and dilute to 1 liter with reagent water. Transfer to a PTFE lined carboy. Using a 1000ml graduated cylinder, transfer an additional 9 liters of reagent water to the carboy. Mix thoroughly. This will prepare 10 liters of Fluid #2. The preparation of all TCLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use. The pH of this solution should be 2.88 $\pm$ 0.05.

NOTE: If the pH of the fluid is out of range, remake the fluid.

5.7 Buffer solution at pH 2, pH 4, pH 7 and pH 10. Commercially available solutions that have been validated by comparison to NIST standards are recommended for routine use. All buffers must be labeled on receipt and after opening. Buffer solutions should be refreshed weekly. **When analyzing samples from West Virginia the buffers must be refreshed on the day of use.**

6.0 Glassware and Apparatus

6.1 Agitation apparatus – Environmental Express, Millipore Corp., or equivalent. Must be capable of rotating the extraction vessels in an end-over-end fashion at 30 $\pm$ 2 rpm.

6.2 Extraction Vessels – 2 liter PTFE coated HDPE bottles

6.3 Filtration device - Millipore Corp.142 mm, or equivalent, capable of exerting pressures of up to 50 psi

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6.4 Filters – Environmental Express or equivalent, 0.7um glass fiber, 142 mm diameter. Filters are acid washed by the manufacturer.

6.5 pH meter - capable of reading ± 0.05 pH units

6.6 Balance - capable of weighing ± 0.01 g

6.7 Graduated cylinders – 100ml and 1000ml

6.8 Beakers – 250 ml glass or plastic

6.9 Watchglass - appropriate to cover beaker

6.10 Magnetic Stirrer and stir bars

6.11 PTFE lined 20 liter carboy

6.12 Thermometer, calibrated against an NIST traceable thermometer

6.13 Water bath – adjustable temperature control

7.0 Procedure

7.1 The preparation of all samples must be documented. See Section 8.1 for the various logbooks and prep sheets that are required for this method. The prep sheet will include such items as: sample ID, bottle number, initial volume, final volume, pHs, lot numbers, batch numbers, and leachate dates and times.

The extraction technician is responsible for filling out all the required information. A copy of the prep sheet will be submitted to the analyst with the leachates. The leaching start date and time are entered into LIMS.

7.2 Determination of Percent Solids

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration, proceed to Section 7.5.

7.2.2 If the sample is liquid or mixed-phase (solid and liquid), proceed as follows. **NOTE:** If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as “totals” instead of TCLP.

7.2.3 Pre-weigh the filter and container that will receive the filtrate. Document all weights in the TCLP_SPLP Description Log.

7.2.4 Assemble the filtering apparatus as per the manufacturer's instructions.

7.2.5 Transfer a 100 gram aliquot of the sample to a beaker and record the weight. If a 100 gram aliquot is not available, inform the Department Supervisor.

7.2.6 Quantitatively transfer the sample aliquot to the filter apparatus. Slurries may be allowed to settle and the liquid portion filtered prior to transferring the solid portion of the sample. **NOTE**: If sample material has adhered to the sample container, obtain the weight of this residue and subtract from the total weight of the sample.

7.2.7 Complete the assembly of the filtration device, and gradually apply pressure until fluid is expelled or 10 psi is obtained. If no fluid is expelled, gradually increase the pressure in 10 psi increments to a maximum of 50 psi. If no fluid is expelled in any 2 minute period, stop the filtration. Shut off the pressurizing gas and vent the filtration system using the top vent.

CAUTION: DO NOT REMOVE FLANGE CLAMPS WHILE SYSTEM IS PRESSURIZED! SERIOUS INJURY MAY RESULT.

NOTE: Instantaneous application of high pressure can cause the filter to clog prematurely.

7.2.8 The material in the filtration apparatus is defined as the solid phase.

NOTE: Some high viscosity liquids (oils, paints) will not filter under these circumstances. The material remaining within the filtration device is defined as the solid phase.

NOTE: If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as “totals” instead of TCLP.

7.2.9 Remove the solid phase of the sample and the filter from the filtration apparatus. If there is a noticeable amount of filtrate entrained in the filter, then dry at 100 °C ± 20 until two successive readings yield the same value within ±1%. Record the final weight.

7.2.10 Determine the percent solids as follows:

$$\% \text{ solids} = \frac{(W - F) \times 100}{T}$$

W = Weight of sample remaining on filter
F = Weight of filter
T = Initial weight of sample used

7.2.11 If the sample contains <0.5% solids, the filtrate is defined as the sample leachate. **NOTE:** Additional aliquots may need to be filtered to generate sufficient volume for all of the analysis. Proceed to Section 7.5.10.

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7.3 Determination of Particle Size

7.3.1 Evaluate the solid portion of the sample for particle size. If the solid portion of sample has a surface area equal to or greater than 3.1 cm^2/gram, then the sample does not require particle size reduction. This would apply to samples such as paper, filter material or rags.

7.3.2 Alternatively, if the solid portion of the sample is smaller than 1 cm in its narrowest dimension (i.e. is capable of passing through a 9.5 mm sieve), then the sample does not require particle size reduction.

7.3.3 If the sample does not meet the particle size criteria listed above, then prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the sample. Care should be taken to minimize any potential metals contamination.

7.3.4 Document the sample description and any particle size reduction in the TCLP_SPLP Description Log.

7.4 Determination of Extraction Fluid

7.4.1 pH Meter Calibration

Make sure that the pH electrode is clean. If the electrode is coated with oil or grease, then it must be washed with a 50% water-methanol solution and then rinse it well with DI water. Do not soak the electrode in the acetone solution. Soak the electrode in a beaker containing pH 7 buffer for approximately 2 hours before using.

Connect the pH electrode to the pH meter. Calibrate the meter using three of the following buffers: pH 2, pH 4, pH 7 or pH 10. Buffers chosen must bracket the pH range. Read back the remaining buffer. For details on specific calibration procedure, see the instruction manual for the meter being used.

After the calibration is complete, analyze the 3 buffer solutions to ensure that an accurate calibration was obtained. Record the results in the TCLP Fluid Determination Log. Readings must be within 0.05 pH units of the buffer solution's true value.

7.4.2 Transfer a 5.0 gram aliquot of the solid phase of the sample to a 250 ml beaker. Record the actual weight in the fluid determination log.

NOTE: The particle size of the solid phase should be < 1mm for this step. This may require some particle size reduction.

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7.4.3 Add 96.5 ml of DI water to the beaker. Place a magnetic stir bar in the beaker and cover with a watch glass. Stir vigorously for 5 minutes. Measure and record the pH in the TCLP Fluid Determination Log. If the pH is <5.0, use extraction fluid #1, and proceed to step 7.5.

7.4.4 If the pH is >5.0, add 3.5 ml of 1.0N HCl and swirl gently. Cover the beaker with a watch glass and heat to 50℃ for 10 minutes.

7.4.5 Allow the solution to cool. Stir vigorously for 5 minutes. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

NOTE: All pH measurements must be recorded to two places after the decimal point.

7.5 Extraction of Semivolatile Organics and Metals

7.5.1 If the sample contains 100% solids, and the sample does not need particle size reduction transfer a minimum of 100g aliquot of the sample to the extraction vessel. Record the weight to ± 0.1 gram on the TCLP_SPLP Sample Prep Sheet. Add 20 times the sample weight of the appropriate extraction fluid to the leaching vessel. Swirl gently and watch for the evolution of carbon dioxide. If no gasses are evolved, cap the container and mount on the rotary agitator. Proceed to section 7.5.4.

7.5.2 If the sample contains <0.5% solids, the filtrate obtained in step 7.2 is defined as the sample leachate. Proceed to section 7.5.10.

7.5.3 If the sample is mixed phase and/or contains >0.5% solids, transfer a 100g aliquot of the sample or more, based on % solids to the filtration device. Assemble the filtration apparatus, and gradually apply pressure to remove any free liquids. Expelled liquid is stored in a glass container, and is to be recombined with sample leachate. Transfer the solid portion of the sample to the appropriate extraction vessel. Add a volume of the appropriate TCLP fluid, calculated as follows:

$$\frac{20 \times \text{sample wt (g)} \times \text{\% solids}}{100} = \text{ml extraction fluid}$$

Swirl gently and watch for evolution of carbon dioxide. Cap the extraction bottle and attach to rotary agitator. Allow the extraction to proceed 18 ± 2 hrs.

NOTE: If the sample contains <25% solids, more sample can be filtered to obtain sufficient solids for leaching such that all analyses may be performed. Consult the Department Supervisor.

7.5.4 Rotate at 30 ±2 rpm. Make sure to measure and record the rotation rate and tumbler ID on the TCLP_SPLP Sample Prep Sheet. Allow the extraction to proceed for 18 ± 2 hrs.

The vessels should be vented periodically during the first hour to prevent pressure build up.

NOTE: The temperature of the extraction room must be 23 ± 2 °C during the extraction period. Record the temperature on TCLP_SPLP Sample Prep Sheet. Use a Hi/Lo thermometer to monitor the room temperature through out the extraction period.

7.5.5 After the leaching period has elapsed, remove the extraction vessels from the rotary agitator and allow them to settle.

7.5.6 Assemble the filtration device. Filter each sample into an appropriately labeled container. Multiple filters may be used. Be sure to thoroughly clean the filtration apparatus between samples.

7.5.7 If a compatible liquid was obtained in Section 7.5.3, combine the liquids at this time.

7.5.8 If the liquids are not compatible, record the total volume of the expelled liquid in the sample description logbook and submit both fractions for analysis. Notify the Department Supervisor.

NOTE: Department Supervisor will notify the Project Manager to determine if the phases are to be mathematically combined as in Section 7.5.13, or to be report separately.

7.5.9 Measure and record the pH of each leachate in the TCLP Fluid Determination Log. See Section 7.4.1 for pH meter calibration.

NOTE: All pH measurements must be recorded to two places after the decimal point.

7.5.10 Aliquot the leachate for the necessary analyses. Listed below are the minimum quantities required for analysis. Additional aliquots will be needed for QC samples.

7.5.10.1	Metals (including Mercury)	100ml
7.5.10.2	Semivolatiles by 8270	100ml
7.5.10.3	Pesticides by 8081	100ml
7.5.10.4	Herbicides by 8151	10ml
7.5.10.5	TPH by 8015	100ml

7.5.11 Leachates for metals analysis are stored in labeled plastic bottles and transferred to the metals department.

7.5.12 All other aliquots (semi-volatile organics) are transferred to amber glass bottles and stored at ≤ 6°C until the subsequent extractions can be performed.

7.5.13 If individual phase are analyzed separately (see 7.5.8) conduct the appropriate analysis and combine the results mathematically by using the following formula:

Final Analyte Conc. = [(V1*C1)+(V2*C2)] / (V1+V2)

V1 = The volume of the first phase (l)
C1 = The conc. of the analyte of concern in the first phase (mg/l)
V2 = The volume of the second phase (l)
C2 = The conc. of the analyte of concern in the second phase (mg/l)

8.0 Documentation

8.1 Documentation for this analysis is quite extensive. At minimum the following logbooks or prep sheets must be filled out completely.

8.1.1 TCLP_SPLP Description Log

8.1.2 TCLP Fluid Determination Log

8.1.3 TCLP_SPLP Sample Prep Sheet

8.1.4 Extraction Bottle Tracking Log

8.1.5 Organics Reagent Log

8.2 For every 20 extractions performed in an extraction vessel, a leachate blank must be performed using that vessel. Document this in the Extraction Bottle Tracking Log.

8.3 For each original matrix type extracted, (soil, water, sludge, etc.) a leachate spike must be performed. Various unique matrices may require their own leachate spikes.

9.0 Quality Assurance, Quality Control and Method Performance

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 6 hours; however, samples should not be added after the QC set has been completed. **NOTE:** All samples and QC samples must leached for the required amount of time.

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9.2 A leachate blank (LB) and sample duplicate (DUP) must be leached with each batch of samples. A leachate blank must be prepared for each fluid type used in a given batch.

9.3 All spiking for the matrix spike (MS) and matrix spike duplicate (MSD), leachate spike (LS), and leachate blank spike (LBS) occurs after filtration.

9.4 Method performance is monitored through the routine analysis of negative and positive control samples. Leachate blank spikes and matrix spikes are not applicable to the leaching portion of this test; however, they will be used later to assess the extraction and digestion efficiency of the specific methods performed on the leachate.

9.5 A sample duplicate is used to assess method precision. Sample duplicate %RPD is compared to method defined control limits. Control limits are stored in the LIMS.

10.0 Safety and Waste Disposal

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling acids, samples, standards or solvents.

10.1.2 Perform all filtration in a fume hood.

10.1.3 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

10.2 Waste Disposal

10.2.1 The TCLP filter and remaining sample is placed in a waste container.

10.2.2 Extra leachate is rinsed down the drain with large amounts of water.

10.2.3 Waste soil from the homogenizing process should be place in the “soil waste” container. **NOTE:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

11.0 References

SW-846 Method 1311, Rev. 0, 07/92

SW-846 Method 1312, Rev. 0, 09/94

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STANDARD OPERATING PROCEDURE FOR THE SYNTHETIC PRECIPITATION LEACHING OF CYANIDE, SEMIVOLATILE ORGANICS, AND METALS (SPLP)

Prepared by:	Norm Farmer	Date:	08/30/13
Reviewed by:	Rick Watkins	Date:	09/13/13

Annual Review

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Reviewed by:	Date:	
Reviewed by:	Date:	

Document Control

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Effective 7 days after "*" date

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TEST NAME: STANDARD OPERATING PROCEDURE FOR THE SYNTHETIC PRECIPITATION LEACHING OF CYANIDE, SEMIVOLATILE ORGANICS, AND METALS (SPLP)

Method: SW846 1312

Dept: OP

Revised Sections: 5.4, 7.1, 7.5.8, and 10.1.3

1.0 Summary, Scope and Application

1.1 Summary

For liquid and aqueous samples containing less than 0.5% solids, the sample is filtered through SPLP filter paper and the filtrate is defined as the SPLP leachate. The leachate can then be analyzed for cyanide, semivolatile organics, and metals.

For solid samples, the solid portion of the sample is extracted by adding extraction fluid equal to 20 times the weight of the sample and rotating the sample for 18 hours at 30 rpm. The extraction fluid used is based on the alkalinity of the solid portion of the sample. After leaching, the sample is filtered through SPLP filter paper. The leachate can then be analyzed for cyanide, semivolatile organics, and metals.

1.2 Scope and Application

This procedure is applicable to samples submitted for SPLP semivolatile analysis and/or SPLP metals analysis.

1.2.1 Metals by 6010

1.2.2 Mercury by 7470

1.2.3 Semivolatiles by 8270

1.2.4 PAHs by 8310 or 8270SIM

1.2.5 Pesticides by 8081

1.2.6 PCBs by 8082

1.2.7 Herbicides by 8151

1.2.8 Extractable TPH by FLPRO or 8015

1.2.9 Cyanide

2.0 Discussion and Comments

This procedure is adapted from SW-846 method 1312. The method utilizes an extraction bottle and rotary agitation device to evaluate the presence and mobility of semivolatile analytes and metals. It is not applicable for evaluating the mobility of volatile analytes.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples. Liquid samples for the analysis of metals only may be collected in HDPE bottles.

3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until leaching.

3.1.3 Samples for SPLP analysis should not be chemically preserved prior to leaching. After filtration, the SPLP leachate for metals should be preserved to a pH <2 with nitric acid unless the leachates will be digested immediately.

3.1.4 SPLP Leachates for semivolatile organics must be protected from light and stored at ≤ 6°C from the time of filtration until extraction.

3.2 Holding Time

3.2.1 Samples submitted for the analysis of semivolatile organics including pesticides and herbicides must be leached within 14 days of collection.

3.2.2 Samples submitted for the analysis cyanide must be leached within 14 days of collection.

3.2.3 Samples submitted for the analysis of mercury must be leached within 28 days of collection.

3.2.4 Samples submitted for the analysis of metals (except mercury) must be leached within 180 days of collection.

3.2.5 Leachates for semivolatile organics must be extracted by the appropriate procedure within 7 days of filtration.

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4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Leachate Blank Spike (LBS): An aliquot of SPLP fluid spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Leachate blank spike recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.8 Leachate Blank (LB): An aliquot of SPLP fluid to which all reagents are added in the same volumes or proportions as used in sample processing. The leachate blank is processed simultaneously with the samples through all the steps of the analytical procedure. The Leachate blank is used to document contamination resulting from the analytical process.

4.9 Leachate Spike (LS): A sample leachate aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The leachate spike recoveries are used to document the bias of a method in a given sample matrix.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

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4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Reagent water – distilled or deionized - free of interferences

5.2 Sulfuric Acid, concentrated, Tracepure or equivalent

5.3 Nitric Acid, concentrated, Tracepure or equivalent

5.4 Sulfuric Acid / Nitric Acid Stock Solution: Add 6.0 g of concentrated Sulfuric acid and 4.0 g of concentrate Nitric acid to a 400 ml beaker and mix thoroughly. Transfer the mixture dropwise to a 500 ml Erlenmeyer flask containing ~300 ml of DI water. Dilute to volume, mix thoroughly, and allow the mixture to cool. The concentration of the mixture is not important as long as the Sulfuric to Nitric ratio remains 60/40. Transfer the mixture to a labeled amber glass bottle and record the information in the Organics Reagent Log.

CAUTION: THIS REAGENT SHOULD BE PREPARED IN A HOOD TO MINIMIZE EXPOSURE TO ACID VAPORS. THE SOLUTION WILL BECOME HOT!

5.5 Extraction Fluid #1: Add a few drops of the acid mixture to a large volume of DI water. Mix thoroughly and check the pH. If the pH is to high, add another drop of the acid mixture. Repeat this procedure until the appropriate pH is reached. The pH of this solution should be 4.20 ± 0.05. The preparation of all SPLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use.

NOTE: This solution is unbuffered and the exact pH may not be attainable.

5.6 Extraction Fluid #2: Add a few drops of the acid mixture to a large volume of DI water. Mix thoroughly and check the pH. If the pH is to high, add another drop of the acid mixture. Repeat this procedure until the appropriate pH is reached. The pH of this solution should be 5.00 ± 0.05. The preparation of all SPLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use.

NOTE: This solution is unbuffered and the exact pH may not be attainable.

5.7 Extraction Fluid #3: The fluid is DI water and is used to evaluate cyanide.

5.8 Buffer solution at pH 4, pH 7 and pH 10. Commercially available solutions that have been validated by comparison to NIST standards are recommended for routine use. All buffers must be labeled on receipt and after opening. Buffer solutions must be refreshed at least weekly.

6.0 Glassware and Apparatus

6.1 Agitation apparatus – Environmental Express, Millipore Corp., or equivalent. Must be capable of rotating the extraction vessels in an end-over-end fashion at 30 ± 2 rpm.

6.2 Extraction Vessels – 2 liter PTFE coated HDPE bottles

6.3 Filtration device - Millipore Corp.142 mm, or equivalent, capable of exerting pressures of up to 50 psi

6.4 Filters – Environmental Express or equivalent, 0.7um glass fiber, 142 mm diameter. Filters are acid washed by the manufacturer.

6.5 pH meter - capable of reading ± 0.05 pH units

6.6 Balance - capable of weighing ± 0.01 g

6.7 Graduated cylinders – 100ml and 1000ml

6.8 Beakers – 250 ml glass or plastic

6.9 PTFE lined 20 liter carboy

6.10 Thermometer, calibrated against an NIST traceable thermometer

7.0 Procedure

7.1 The preparation of all samples must be documented. See Section 8.1 for the various logbooks and prep sheets that are required for this method. The prep sheet will include such items as: sample ID, bottle number, initial volume, final volume, pHs, lot numbers, batch numbers, and leachate dates and times.

The extraction technician is responsible for filling out all the required information. A copy of the prep sheet will be submitted to the analyst with the leachates. The leaching start date and time are entered into LIMS.

7.2 Determination of Percent Solids

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration, proceed to Section 7.5.

7.2.2 If the sample is liquid or mixed-phase (solid and liquid), proceed as follows. **NOTE:** If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as "totals" instead of SPLP.

7.2.3 Pre-weigh the filter and container that will receive the filtrate. Document all weights in the TCLP_SPLP Description Log.

7.2.4 Assemble the filtering apparatus as per the manufacturer's instructions.

7.2.5 Transfer a 100 gram aliquot of the sample to a beaker and record the weight. If a 100 gram aliquot is not available, inform the Department Supervisor.

7.2.6 Quantitatively transfer the sample aliquot to the filter apparatus. Slurries may be allowed to settle and the liquid portion filtered prior to transferring the solid portion of the sample. **NOTE**: If sample material has adhered to the sample container, obtain the weight of this residue and subtract from the total weight of the sample.

7.2.7 Complete the assembly of the filtration device, and gradually apply pressure until fluid is expelled or 10 psi is obtained. If no fluid is expelled, gradually increase the pressure in 10 psi increments to a maximum of 50 psi. If no fluid is expelled in any 2 minute period, stop the filtration. Shut off the pressurizing gas and vent the filtration system using the top vent.

CAUTION: DO NOT REMOVE FLANGE CLAMPS WHILE SYSTEM IS PRESSURIZED! SERIOUS INJURY MAY RESULT.

NOTE: Instantaneous application of high pressure can cause the filter to clog prematurely.

7.2.8 The material in the filtration apparatus is defined as the solid phase.

NOTE: Some high viscosity liquids (oils, paints) will not filter under these circumstances. The material remaining within the filtration device is defined as the solid phase.

7.2.9 Remove the solid phase of the sample and the filter from the filtration apparatus. If there is a noticeable amount of filtrate entrained in the filter, then dry at 100 °C ± 20 until two successive readings yield the same value within ±1%. Record the final weight.

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7.2.10 Determine the percent solids as follows:

$$\% \text{ solids} = \frac{(W - F) \times 100}{T}$$

W = Weight of sample remaining on filter
F = Weight of filter
T = Initial weight of sample used

7.2.11 If the sample contains <0.5% solids, the filtrate is defined as the sample leachate. **NOTE:** Additional aliquots may need to be filtered to generate sufficient volume for all of the analysis. Proceed to Section 7.5.10.

7.3 Determination of Particle Size

7.3.1 Evaluate the solid portion of the sample for particle size. If the solid portion of sample has a surface area equal to or greater than 3.1 cm^2/gram, then the sample does not require particle size reduction. This would apply to samples such as paper, filter material or rags.

7.3.2 Alternatively, if the solid portion of the sample is smaller than 1 cm in its narrowest dimension (i.e. is capable of passing through a 9.5 mm sieve), then the sample does not require particle size reduction.

7.3.3 If the sample does not meet the particle size criteria listed above, then prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the sample. Care should be taken to minimize any potential metals contamination.

7.3.4 Document the sample description and any particle size reduction in the TCLP_SPLP Description Log.

7.4 Determination of Extraction Fluid

7.4.1 pH Meter Calibration

Make sure that the pH electrode is clean. If the electrode is coated with oil or grease, then it must be washed with a 50% water-methanol solution and then rinse it well with DI water. Do not soak the electrode in the acetone solution. Soak the electrode in a beaker containing pH 7 buffer for approximately 2 hours before using.

Connect the pH electrode to the pH meter. Calibrate the meter using two points, pH 4 and pH 7 or pH 4 and pH 10, stirring gently. Read the remaining buffer. **NOTE:** For details on specific calibration procedure, see the instruction manual for the meter being used.

After the calibration is complete, analyze the 3 buffer solutions to ensure that an accurate calibration was obtained. Record the results on the Fluid

determination worksheet. Readings must be within 0.05 pH units of the buffer solution's true value.

7.4.2 Fluid determination for SPLP is based on the matrix, analysis to be performed, and location of where the samples were collected. Document the fluid to be used in the SPLP Fluid Determination Log.

7.4.3 Fluid #1 is used for the analysis of **soil** and **sediment** samples that were collected **East of the Mississippi River (See TABLE 1).** It is used to evaluate **metals** and **semivolatile organics**.

7.4.4 Fluid #1 is also used for the analysis of all **waste** and **wastewater** samples. It is used to evaluate **metals** and **semivolatile organics**.

7.4.5 Fluid #2 is used for the analysis of **soil** and **sediment** samples that were collected **West of the Mississippi River (See TABLE 1).** It is used to evaluate **metals** and **semivolatile organics**.

7.4.6 Fluid #3 is used for the analysis of all cyanide containing samples. It is used to evaluate **cyanide** only.

7.5 Extraction of Semivolatile Organics and Metals

7.5.1 If the sample contains 100% solids, and the sample does not need particle size reduction transfer a minimum of 100g aliquot of the sample to the extraction vessel. Record the weight to ± 0.1 gram on the TCLP_SPLP Sample Prep Sheet. Add 20 times the sample weight of the appropriate extraction fluid to the leaching vessel. Swirl gently and watch for the evolution of carbon dioxide. If no gasses are evolved, cap the container and mount on the rotary agitator. Proceed to section 7.5.4.

7.5.2 If the sample contains <0.5% solids, the filtrate obtained in step 7.2 is defined as the sample leachate. Proceed to section 7.5.10.

7.5.3 If the sample is mixed phase and/or contains >0.5% solids, transfer a 100g aliquot of the sample or more, based on % solids to the filtration device. Assemble the filtration apparatus, and gradually apply pressure to remove any free liquids. Expelled liquid is stored in a glass container, and is to be recombined with sample leachate. Transfer the solid portion of the sample to the appropriate extraction vessel. Add a volume of the appropriate SPLP fluid, calculated as follows:

$$\frac{20 \times \text{sample wt (g)} \times \text{\% solids}}{100} = \text{ml extraction fluid}$$

Swirl gently and watch for evolution of carbon dioxide. Cap the extraction bottle and attach to rotary agitator. Allow the extraction to proceed 18 ± 2 hrs.

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NOTE: If the sample contains <25% solids, more sample can be filtered to obtain sufficient solids for leaching such that all analyses may be performed. Consult the Department Supervisor.

7.5.4 Rotate at 30 ±2 rpm. Make sure to measure and record the rotation rate on the TCLP_SPLP Sample Prep Sheet. Allow the extraction to proceed for 18 ± 2 hrs.

The vessels should be vented periodically during the first hour to prevent pressure build up.

NOTE: The temperature of the extraction room must be 23 ± 2 °C during the extraction period. Record the temperature on TCLP_SPLP Sample Prep Sheet. Use a Hi/Lo thermometer to monitor the room temperature through out the extraction period.

7.5.5 After the leaching period has elapsed, remove the extraction vessels from the rotary agitator and allow them to settle.

7.5.6 Assemble the filtration device. Filter each sample into an appropriately labeled container. Multiple filters may be used. Be sure to thoroughly clean the filtration apparatus between samples.

7.5.7 If a compatible liquid was obtained in Section 7.5.3, combine the liquids at this time.

7.5.8 If the liquids are not compatible, record the total volume of the expelled liquid in the sample description logbook and submit both fractions for analysis. Notify the Department Supervisor.

NOTE: Department Supervisor will notify the Project Manager to determine if the phases are to be mathematically combined as in Section 7.5.13, or to be report separately.

7.5.9 Measure and record the pH of each leachate in the SPLP Fluid Determination Log. See Section 7.4.1 for pH meter calibration.

NOTE: All pH measurements must be recorded to two places after the decimal point.

7.5.10 Aliquot the leachate for the necessary analyses. Listed below are the minimum quantities required for analysis. Additional aliquots will be needed for QC samples.

7.5.10.1	Metals (including Mercury)	200ml
7.5.10.2	Semivolatiles by 8270	1000ml
7.5.10.3	PAHs by 8310 or 8270SIM	1000ml
7.5.10.4	Pesticides by 8081	1000ml

7.5.10.5	PCBs by 8082	1000ml
7.5.10.6	Herbicides by 8151	100ml
7.5.10.7	TPH by FLPRO	100ml
7.5.10.8	TPH by 8015	100ml
7.5.10.9	Cyanide	100ml

7.5.11 Leachates for metals analysis are stored in labeled plastic bottles and transferred to the metals department.

7.5.12 Leachates for cyanide analysis are stored in labeled plastic bottles and transferred to the general chemistry department.

7.5.13 All other aliquots (semi-volatile organics) are transferred to amber glass bottles and stored at ≤ 6°C until the subsequent extractions can be performed.

7.5.14 If individual phase are analyzed separately (see 7.5.8) conduct the appropriate analysis and combine the results mathematically by using the following formula:

Final Analyte Conc. = [(V1*C1)+(V2*C2)] / (V1+V2)

V1 = The volume of the first phase (l)
C1 = The conc. of the analyte of concern in the first phase (mg/l)
V2 = The volume of the second phase (l)
C2 = The conc. of the analyte of concern in the second phase (mg/l)

8.0 Documentation

8.1 Documentation for this analysis is quite extensive. At minimum the following logbooks or prep sheets must be filled out completely.

8.1.1 TCLP_SPLP Description Log

8.1.2 SPLP Fluid Determination Log

8.1.3 TCLP_SPLP Sample Prep Sheet

8.1.4 Extraction Bottle Tracking Log

8.1.5 Organics Reagent Log

8.2 For every 20 extractions performed in an extraction vessel, a leachate blank must be performed using that vessel. Document this in the Extraction Bottle Tracking Log.

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8.3 For each original matrix type extracted, (soil, water, sludge, etc.) a leachate spike must be performed. Various unique matrices may require their own leachate spikes.

9.0 Quality Assurance, Quality Control and Method Performance

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 6 hours; however, samples should not be added after the QC set has been completed. **NOTE:** All samples and QC samples must leached for the required amount of time.

9.2 A leachate blank (LB) and sample duplicate (DUP) must be leached with each batch of samples. A leachate blank must be prepared for each fluid type used in a given batch.

9.3 All spiking for the matrix spike (MS) and matrix spike duplicate (MSD), leachate spike (LS), and leachate blank spike (LBS) occurs after filtration.

9.4 Method performance is monitored through the routine analysis of negative and positive control samples. Leachate blank spikes and matrix spikes are not applicable to the leaching portion of this test; however, they will be used later to assess the extraction and digestion efficiency of the specific methods performed on the leachate.

9.5 A sample duplicate is used to assess method precision. Sample duplicate %RPD is compared to method defined control limits. Control limits are stored in the LIMS.

10.0 Safety and Waste Disposal

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling acids, samples, standards or solvents.

10.1.2 Perform all filtration in a fume hood.

10.1.3 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

10.2 Waste Disposal

10.2.1 The SPLP filter and remaining sample is placed in a waste container.

10.2.2 Extra leachate is rinsed down the drain with large amounts of water.

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10.2.3 Waste soil from the homogenizing process should be place in the "soil waste" container. **NOTE:** Waste soil from foreign soils must follow "foreign soil" disposal requirements.

11.0 References

SW-846 Method 1311, Rev. 0, 07/92

SW-846 Method 1312, Rev. 0, 09/94

TABLE 1

Fluid #1		Fluid #2	
State	**Abrev.**	**State**	**Abrev.**
Alabama	AL	Alaska	Ak
Connecticut	CT	Arizona	AZ
Delaware	DE	Arkansas	AR
District of Columbia	DC	California	CA
Florida	FL	Colorado	CO
Georgia	GA	Hawaii	HI
Illinois	IL	Idaho	ID
Indiana	IN	Iowa	IA
Kentucky	KY	Kansas	KS
Maine	ME	Lousiana	LA
Maryland	MD	Minnesota	MN
Massachusetts	MA	Missouri	MO
Michigan	MI	Montana	MT
Mississippi	MS	Nebraska	NE
New Hampshire	NH	Nevada	NV
New Jersey	NJ	New Mexico	NM
New York	NY	North Dakota	ND
North Carolina	NC	Oklahoma	OK
Ohio	OH	Oregon	OR
Pennsylvania	PA	South Dakota	SD
Puerto Rico	PR	Texas	TX
Rhode Island	RI	Utah	UT
South Carolina	SC	Washington	WA
Tennessee	TN	Wyoming	WY
Vermont	VT		
Virginia	VA		
West Virginia	WV		
Wisconsin	WI		

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Quality Systems Manual

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INTRODUCTION

The Accutest Laboratories Southeast, Inc. (Accutest SE) Quality Assurance Program, detailed in this plan, has been designed to meet the quality program requirements of the National Environmental Laboratories Accreditation Conference (TNI), DoD QSM Ver 4.2, 2010 and ISO 17025. The plan establishes the framework for documenting the requirements of the quality processes regularly practiced by the Laboratory. The Quality Assurance Officer is responsible for changes to the Quality Assurance Program, which are appended to the LQSM as they occur. The plan is reviewed annually for compliance purposes by the Laboratory Director and Technical Director and edited if necessary. Changes that are incorporated into the plan are summarized in the plan introduction. Changes to the plan are communicated to the general staff in a meeting conducted by the Quality Assurance Officer following the plan's approval.

The Accutest SE plan is supported by standard operating procedures (SOPs), which provide specific operational instructions on the execution of each quality element and assure that compliance with the requirements of the plan are achieved. Accutest SE employees are responsible for knowing the requirements of the SOPs and applying them in the daily execution of their duties. These documents are updated as changes occur and the staff is trained to apply the changes.

At Accutest, we believe that satisfying client requirements and providing a product that meets or exceeds the standards of the industry is the key to a good business relationship. However, client satisfaction cannot be guaranteed unless there is a system that assures the product consistently meets its design requirements and is adequately documented to assure that all procedural steps are executed and are traceable.

This plan has been designed to assure that this goal is consistently achieved and the Accutest product withstands the rigors of scrutiny that are routinely applied to analytical data and the processes that support its generation.

Accutest Laboratories Southeast is a permanent location facility and is part of Accutest Laboratories, Inc.

Summary of Changes
Accutest SE Quality System Manual –October 2012

Section	**Description**	Page #
Title Page	new revision number	Title
OrgChart	Lillian Torres replaced with Angel Rivera as WetChem supervisor; removed Paul Konnik from Sales.	8
1	Management commitment ro constant process improvement spelled out	5
16	Complete rewrite with detail and hierarchy of non-conforming products	63
App II	DoD certified methods specified in both TNI and non-TNI tables Added Perchlorate, Nitrate/Nitrite, 1,4-Dioxane,	80-83
App IV	Added 2 MS SOPs and 1 Sample Management SOP	99-101

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1.0 QUALITY POLICY

1.1 ***Accutest Mission***:

Accutest Laboratories provides analytical services to commercial and government clients in support of environmental monitoring and remedial activities as requested. The Laboratory's mission is dedicated to providing reliable data that satisfies clients requirements as explained in the following: "Provide easy access, high quality, analytical support to commercial and government clients which meet or exceeds data quality objectives and provides them with the data needed to satisfy regulatory requirements and/or make confident decisions on the effectiveness of remedial activities."

These services are provided impartially and are not influenced by undue commercial or financial pressures, which might impact the staff's technical judgment. Coincidentally, Accutest does not engage in activities that endanger the trust in our independent judgment and integrity in relation to the testing activities performed.

1.2 ***Policy Statement*:**

The management and staff of Accutest Laboratories share the responsibility for product quality and continually strive for its systematic improvement. Accordingly, Accutest's quality assurance program is designed to assure that all processes and procedures, which are components of environmental data production, meet established industry requirements, are adequately documented from a procedural and data traceability perspective, and are consistently executed by the staff. It also assures that analytical data of known quality, meeting the quality objectives of the analytical method in use and the data user's requirements, is consistently produced in the laboratory. This assurance enables the data user to make rational, confident, cost-effective decisions on the assessment and resolution of environmental issues.

The laboratory Quality System also provides the management staff with data quality and operational feedback information. This enables them to determine if the laboratory is achieving the established quality and operational standards, which are dictated by the client or established by regulation, such as TNI, ISO 17025 or DoD QSM. The information provided to management, through the QA program, is used to assess operational performance from a quality perspective and to perform corrective action as necessary.

All employees of Accutest Laboratories participating in environmental testing receive quality system training and are responsible for knowing and complying with the system requirements. The entire staff shares Accutest's commitment to good professional practice.

Harry Behzadi, Ph.D.
VP Southeast Operations

2.0 ORGANIZATION

2.1 ***Organizational Entity***. Accutest Laboratories, Inc. is a testing laboratory founded in 1956 and registered as a New Jersey Corporation. In 2007 the laboratory has changed ownership to Accutest Holdings, Inc. Operations, staff and physical locations were not affected by the change. The laboratory headquarters are located in Dayton, New Jersey where it has conducted business since 1987. Satellite laboratories are maintained in Marlborough, Massachusetts; Orlando, Florida; San Jose, California; Denver, Colorado; Lafayette, Louisiana; and Houston, Texas.

2.2 ***Management Responsibilities***

Requirement: Each laboratory facility will have an established chain of command. The duties and responsibilities of the management staff are linked to the President/CEO of Accutest Laboratories who establishes the agenda for all company activities.

President/CEO*.* Primarily responsible for all operations and business activities. Delegates authority to laboratory directors, general managers, and quality assurance director to conduct day-to-day operations and execute quality assurance duties. Each of the individual operational entities (New Jersey, Massachusetts, Florida,, Texas, California, Colorado, and Louisiana) reports to the President/CEO.

Corporate Quality Assurance Director*.* Responsible for design, oversight, and facilitation of all quality assurance activities established by the Quality Program. Directly reports to the President/CEO.

Vice President Operations/Laboratory Director*.* There is a Laboratory Director assigned to each of the following operational entities: New Jersey, Massachusetts Florida, Louisiana, and West (Texas, California, and Colorado). The Laboratory Director executes day-to-day responsibility for laboratory operations including technical aspects of production activities and associated logistical procedures. Directly reports to the President/CEO.

Quality Assurance Officer *(on location).* Responsible for oversight, implementation and facilitation of all quality assurance activities established by the Quality Program. Directly reports to the Laboratory Director. Also exchanges information with and submits laboratory performance data (PE scores, audit reports, accreditation changes, etc.) to Corporate QA Director. Takes program directions from Corporate QA Director.

Technical Director. Responsible for oversight and implementation of technical aspects of production activities in the environmental testing laboratory. In the event that the technical director, quality assurance director, or laboratory manager is absent for a period of time that exceeds 15 consecutive calendar days, the designated appointees shall temporarily perform the technical director, quality assurance director, or laboratory manager's job function. If this absence exceeds 65 consecutive calendar days, the Accreditation Body(ies), including DoD ELAP, is to be notified in writing.

Current list of appointed deputies located in restricted access controlled document directory

Department Managers. Executes day-to-day responsibility for specific laboratory areas including technical aspects of production activities and associated logistical procedures. Directly report to the Laboratory Director.

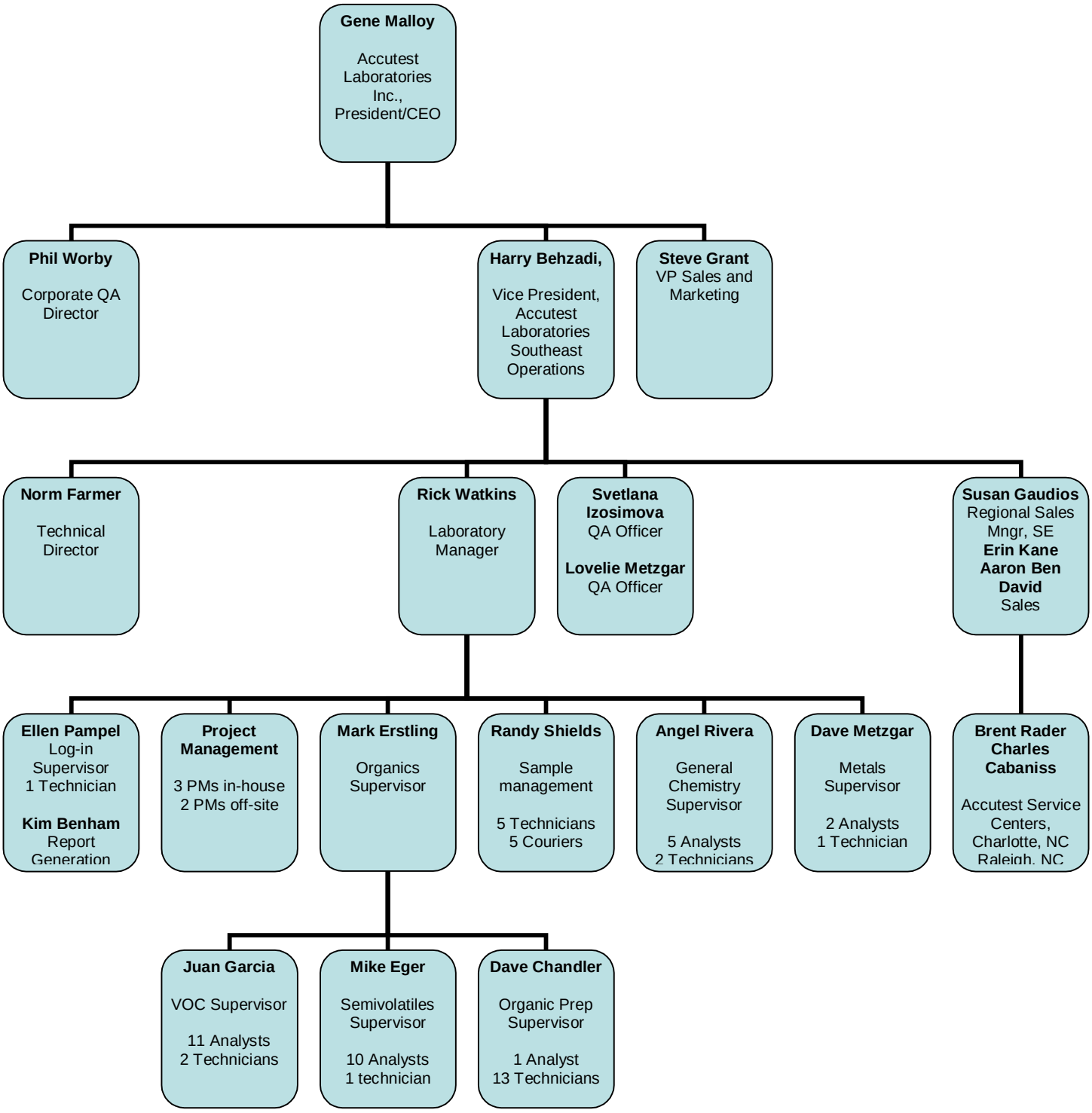
Section Supervisors. Executes day-to-day responsibility for specific laboratory units including technical aspects of production activities and associated logistical procedures. Directly report to the Department Manager.

2.3 ***Chain of Command***

The responsibility for managing all aspects of the Company's operation is delegated to specific individuals, who have been assigned the authority to act in the absence of the senior staff. These individuals are identified in the following Chain of Command:

Harry Behzadi, Ph.D., VP, Southeast Operations
Norm Farmer, Technical Director (Operations and IT)
Rick Watkins, Laboratory Manager (Operations)
Heather Wandrey, Project Manager (Client Services)

Accutest Laboratories Southeast Organizational Chart



3.0 QUALITY RESPONSIBILITIES OF THE MANAGEMENT TEAM

3.1 ***Requirement*:** Each member of the management team has a defined responsibility for the Quality Program. Program implementation and operation is designated as an operational management responsibility. Program design and implementation is designated as a Quality Assurance Responsibility.

President/CEO*:* Primary responsibility for all quality activities. Delegates program responsibility to the Quality Assurance Director. Serves as the primary alternate in the absence of the Quality Assurance Director. Has the ultimate responsibility for implementation of the Quality Program.

Vice President Operations/Laboratory Director*.* Responsible for implementing and operating the Quality Program in all laboratory areas. Responsible for the design and implementation of corrective action for defective processes. Has the authority to delegate Quality Program implementation responsibilities.

Corporate Quality Assurance Director*.* Responsible for design, implementation support, training, and monitoring of the quality system. Identifies product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Quality Assurance Officer *(on location).* Responsible for design support, implementation support, and monitoring support of the quality system. Training personnel in various aspects of quality system. Conducts audits and product reviews to identify product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Technical Director. Responsible for oversight and implementation of technical aspects of Quality System as they are integrated into method applications and employed to assess analytical controls on daily basis. The Technical Director reviews and acknowledges the technical feasibility of proposed quality system involving technical applications.

Department Managers*.* Responsible for applying the requirements of the Quality Program in their section and assuring subordinate supervisors and staff apply all program requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Group Leaders*.* Responsible for applying the requirements of the Quality Program to their operation and assuring the staff applies all program requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Bench Analysts. Responsible for applying the requirements of the Quality Program to the analyses they perform, evaluating QC data and initiating corrective action for quality control deficiencies within their control. Implements global corrective action as directed by superiors.

3.2 ***Program Authority***:

Authority for program implementation on corporate level originates with the President/CEO who bears ultimate responsibility for program design, implementation, and enforcement of requirements. This authority and responsibility is delegated to the Director of Quality Assurance who performs quality functions independently without the encumbrances or biases created by operational or production responsibilities to ensure an honest, independent assessment of quality issues.

Laboratory Director and Quality Assurance Officer mirror this authority on location.

3.3 ***Data Integrity Policy***:

The Accutest Data Integrity Policy reflects a comprehensive, systematic approach for assuring that data produced by the laboratory accurately reflects the outcome of the tests performed on field samples and has been produced in a bias free environment by ethical professionals. The policy includes a commitment to technical ethics, staff training in ethics and data integrity, an individual attestation to data integrity and procedures for evaluating data integrity. Senior management assumes the responsibility for assuring compliance with all technical ethics elements and operation of all data integrity procedures. The staff is responsible for compliance with the ethical code of conduct and for practicing data integrity procedures.

The Accutest Data Integrity Policy is as follows:

"Accutest Laboratories is committed to producing data that meets the data integrity requirements of the environmental regulatory community. This commitment is demonstrated through the application of a comprehensive data integrity program that includes ethics and data integrity training, data integrity evaluation procedures, staff participation and management oversight. Adherence to the specifications of the program assures that data provided to our clients is of the highest possible integrity and can be used for decision making processes with high confidence."

Data Integrity Responsibilities

***Management*.** Senior management retains oversight responsibility for the data integrity program and retains ultimate responsibility for execution of the data integrity program elements. Senior management is responsible for providing the resources required to conduct ethics training and operate data integrity evaluation procedures. They also include responsibility for creating an environment of trust among the staff and being the lead advocate for promoting the data integrity policy and the importance of technical ethics.

***Staff*.** The staff is responsible for adhering to the company ethics policy as they perform their duties and responsibilities associated with sample analysis and reporting. By executing this responsibility, data produced by Accutest Laboratories retains its high integrity characteristics and withstands the rigors of all data integrity checks.

The staff is also responsible for adhering to all laboratory requirements pertaining to manual data edits, data transcription and data traceability. These include the application of approved manual peak integration and documentation procedures. It also includes establishing traceability for all manual results calculations and data edits.

***Ethics Statement*.** The Accutest ethics statement reflects the standards that are expected for businesses that provide environmental services to regulated entities and regulatory agencies on a commercial basis. The Ethics Policy is comprised of key elements that are essential to organizations that perform chemical analysis for a fee. As such, it focuses on elements related to personal, technical and business activities.

Accutest Laboratories provides analytical chemistry services on environmental matters to the regulated community. The data the company produces provides the foundation for determining the risk presented by a chemical pollutant to human health and the environment. The environmental industry is dependent upon the accurate portrayal of environmental chemistry data. This process is reliant upon a high level of scientific and personal ethics.

It is essential to the Company that each employee understands the ethical and quality standards required to work in this industry. Accordingly, Accutest has adopted a code of ethics, which each employee is expected to adhere to as follows:

- Perform chemical and microbiological analysis using accepted scientific practices and principles.
- Perform tasks in an honest, principled and incorruptible manner inspiring peers & subordinates.
- Maintain professional integrity as an individual.
- Provide services in a confidential, honest, and forthright manner.
- Produce results that are accurate and defensible.

- Report data without any considerations of self-interest.

- Comply with all pertinent laws and regulations associated with assigned tasks and responsibilities.

Data Integrity Procedures.

Four key elements comprise the Accutest data integrity system:
1) data integrity training,
2) signed data integrity documentation for all laboratory employees,
3) in-depth, periodic monitoring of data integrity, and
4) data integrity procedure documentation.
Procedures have been implemented for conducting data integrity training and for documenting that employees conform to the Accutest Data Integrity and Ethics policy.

The data integrity program consists of routine data integrity evaluation and documentation procedures to periodically monitor and document data integrity. These procedures are documented in SOPs. SOPs are approved and reviewed annually following the procedures employed for all Accutest SOPs. Documentation associated with data integrity evaluations is maintained on file and is available for review.

Data Integrity Training, .Accutest employees receive technical ethics training during new employee orientation. Employees are also required to attend annual ethics refreshment training and sign an ethical conduct agreement annually, which verifies their understanding of Accutest's technical ethics policy and their ethical responsibilities. The agreement is refreshed annually and appended to each individual's training file.

The training focuses on the reasons for technical ethic training, explains the impact of data fraud on human health and the environment, and illustrates the consequences of criminal fraud on businesses and individual careers. Multiple examples of prohibited practices are reviewed and discussed. Accutest's ethics policy and code of ethics are reviewed and explained for each new employee. Employees receive Accutest's technical ethics brochure for further review.

Training on department-specific data integrity procedures are conducted by individual departments for groups involved in data operations. These include procedures for manual chromatographic peak integration, standards traceability, etc.

Data Integrity Training Documentation. Records of all data integrity training are maintained in individual training folders. Attendance at all training sessions is documented and appended to the training file.

Accutest Data Integrity and Ethical Conduct Agreement. All employees are required to sign a Data Integrity and Ethical Conduct Agreement annually. This document is archived in individual training files, which are retained for duration of employment.

The Data Integrity and Ethical Conduct Agreement is as follows**:**

I. *I understand the high ethical standards required of me with regard to the duties I perform and the data I report in connection with my employment at Accutest Laboratories.*

II. *I have received formal instruction on the code of ethics that has been adapted by Accutest Laboratories and agree to comply with these requirements.*

III. *I have received formal instruction on the elements of Accutest Laboratories' Data Integrity Policy and have been informed of the following specific procedures:*

   a. *Routine data integrity monitoring is conducted on sample data, which may include an evaluation of the data I produce,*

   b. *Formal procedures for the confidential reporting of data integrity issues are available, which can be used by any employee,*

   c. *A data integrity investigation is conducted when data issues are identified that may negatively impact data integrity.*

IV. *I am aware that data fraud is a punishable crime that may include fines and/or imprisonment upon conviction.*

V. *I also agree to the following:*

   a. *I shall not intentionally report data values, which are not the actual values observed or measured.*

   b. *I shall not intentionally modify data values unless the modification can be technically justified through a measurable analytical process.*

   c. *I shall not intentionally report dates and times of data analysis that are not the true and actual times the data analysis was conducted.*

   d. *I shall not condone any accidental or intentional reporting of inauthentic data by other employees and immediately report it's occurrence to my superiors.*

   e. *I shall immediately report any accidental reporting of inauthentic data by myself to my superiors.*

Data Integrity Monitoring. Several documented procedures are employed for performing data integrity monitoring. These include regular data review procedures by supervisory and management staff (Section 12.7), supervisory review and approval of manual integrations and periodic reviews of data audit trails from the LIMS and all computer controlled analysis.

Data Review. All data produced by the laboratory undergoes several levels of review, which includes two levels of management review. Detected data anomalies that appear to be related to data integrity issues are isolated for further investigation. The investigation is conducted following the procedures described in this section.

Manual Peak Integration Review and Approval. Routine data review procedures for all chromatographic processes includes a review of all manual chromatographic peak integrations. This review is performed by the management staff and consists of a review of the machine integration compared to the manual integration. Manual integrations, which have been performed in accordance with Accutest's manual peak integration procedures are approved for further processing and release. Manual integrations which are not performed to Accutest's specifications are set aside for corrective action, which may include analyst retraining or further investigation as necessary.

Data Audit Trail Review. Data integrity audits are comprehensive data package audits that include a review of raw data, process logbooks, processed data reports and data audit trails from individual instruments and LIMS. Data audit trails, which record all electronic data activities, are available for the majority of computerized methodology and the laboratory information management system (LIMS). These audit trails are periodically reviewed to determine if interventions performed by technical staff constitute an appropriate action. The review is performed on a recently completed job and includes interviews with the staff that performed the analysis. Findings indicative of inappropriate interventions or data integrity issues are investigated to determine the cause and the extent of the anomaly.

Confidential Reporting Of Data Integrity Issues. Data integrity concerns may be raised by any individual to their supervisor. Employees with data integrity concerns should always discuss those concerns with their immediate supervisors as a first step unless the employee is concerned with the confidentiality of disclosing data integrity issues or is uncomfortable discussing the issue with their immediate supervisors. The supervisor makes an initial assessment of the situation to determine if the concern is related to a data integrity violation. Those issues that appear to be violations are documented by the supervisor and referred to the QA Officer (local) for investigation.

Documented procedures for the confidential reporting of data integrity issues in the laboratory are part of the data integrity policy. These procedures assure that laboratory staff can privately discuss ethical issues or report items of ethical concern without fears of repercussions with senior staff.

Employees with data integrity concerns that they consider to be confidential are directed to the Corporate Human Resources Manager in Dayton, New Jersey. The HR Manager acts as a conduit to arrange a private discussion between the employee and the Corporate QA Director or a local QA Officer.

During the employee - QA discussion, the QA representative evaluates the situation presented by the employee to determine if the issue is a data integrity concern or a legitimate practice. If the practice is legitimate, the QA representative clarifies the

process for the employee to assure understanding. If the situation appears to be a data integrity concern, the QA representative initiates a Data Integrity Investigation following the procedures specified in SOPs QA038-QA041.

Data Integrity Investigations. Follow-up investigations are conducted for all reported instances of ethical concern related to data integrity. Investigations are performed in a confidential manner by senior management according to a documented procedure. The outcome of the investigation is documented and reported to the company president who has the ultimate responsibility for determining the final course of action in the matter. Investigation documentation includes corrective action records, client notification information and disciplinary action outcomes, which is archived for a period of five years.

The investigations are conducted by the senior staff and supervisory personnel from the affected area. The investigation team includes the Laboratory Director and the Quality Assurance Officer. Investigations are conducted in a confidential manner until it is completed and resolved.

The investigation includes a review of the primary information in question by the investigations team. The team performs a review of associated data and similar historical data to determine if patterns exist. Interviews are conducted with key staff to determine the reasons for the observed practices.

Following data compilation, the investigations team reviews all information to formulate a consensus conclusion. The investigation results are documented along with the recommended course of action.

Corrective Action, Client Notification & Discipline. Investigations that reveal systematic data integrity issues will go through corrective action for resolution and disposition (Section 13). If the investigation indicates that an impact to data has occurred and the defective data has been released to clients, client notification procedures will be initiated following the steps in Section 17.6.

In all cases of data integrity violations, some level of disciplinary action will be conducted on the responsible individual. The level of discipline will be consistent with the violation and may range from retraining and/or verbal reprimand to termination.

4.0 JOB DESCRIPTIONS OF KEY STAFF

4.1 ***Requirement*:** Descriptions of key positions within the organization must be defined to ensure that clients and staff understand duties and the responsibilities of the management staff and the reporting relationships between positions.

President/Chief Executive Officer. Responsible for all laboratory operations and business activities. Establishes the company mission and objectives in response to business needs. Direct supervision of the Vice President of Operations, each laboratory director, client services, management information systems, and quality assurance.

Vice President, Operations/Laboratory Director. Reports to the company president. Establishes regional laboratory operations strategy and business development. Authorized to enter into contractual agreements on Company's behalf.

Director, Quality Assurance. Reports to the company president. Establishes the company quality agenda, develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities monitors the quality system and provides quality system feedback to management to be used for process improvement.

Vice President, Information Technlogies Reports to the company president. Develops the MIS software and hardware agenda. Provides system strategies to compliment company objectives. Maintains all software and hardware used for data handling.

Client Services, Sales, Account Manager(s). Reports to the company president. Establishes and maintains communications between clients and the laboratory pertaining to client requirements which are related to sample analysis and data deliverables. Initiates client orders and supervises sample login operations.

Quality Assurance Officer *(on location)*. Reports to the Laboratory Director. Develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities, monitors the quality system, and provides quality system feedback to management to be used for process improvement. In the event of prolonged absence QAO also designated a Deputy Technical Director, unless otherwise specified by internal memo from Laboratory Director.

Manager Client Services *(on location)*. Reports to the Laboratory Director. Establishes and maintains communications between clients and the laboratory pertaining to client requirements which are related to sample analysis and data deliverables. Initiates client orders and supervises sample login operations.

Technical Director (On Location). Reports to the laboratory director. Establishes laboratory operations strategy. Direct supervision of organic chemistry and inorganic

chemistry. Directs the operations, preparation and instrumental analysis. Responsible for following Quality Program requirements. Assumes operational responsibilities of Lab Director in his absence.

Laboratory Manager. Reports to the Laboratory Director. Directs the day-to day operations of entire laboratory, direct supervision of organic chemistry, inorganic chemistry, field services, and sample management.
Oversees daily work schedule as developed by respective departments. Supervises method implementation. Responsible for following Quality Program requirements. Maintains laboratory instrumentation in an operable condition.

Supervisors, Shipping and Receiving Departments. Reports to the Laboratory Manager. Develops, maintains and executes all procedures required for transport and receipt of samples, verification of preservation, and chain of custody documentation. Responsible for maintaining and documenting secure storage, delivery of samples to laboratory units on request, and disposal following completion of all analytical procedures.

Supervisor, Wet Chemistry. Reports to the Laboratory Manager. Directs the operations of the wet chemistry group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for wet chemistry parameters using valid, documented methodology. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality Program requirements.

Supervisor, Metals. Reports to the Laboratory Manager. Directs the operations of the metals group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for metallic elements using valid, documented methodology. Documents all procedures and data production activities. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality Program requirements

Supervisor, Organic Preparation. Reports to the Laboratory Manager. Directs the operations of the sample preparation group. Establishes and executes daily work schedule. Supervises method implementation, and application. Supervises the preparation of samples for organic compounds using valid, documented methodology. Documents all procedures and data production activities. Maintains laboratory equipment in an operable condition. Reviews records for compliance to quality and methodological requirements. Responsible for following Quality Program requirements.

Volatile and Semivolatie Supervisors, Organics. Reports to the Laboratory Manager. Directs the operations of the respective organics group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for organic compounds using valid, documented methodology. Documents all procedures and data production

activities. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality Program requirements

Report Generation Supervisor. Reports to Laboratory Manager. Oversees report generation and fulfillment of client specifications as applied to data deliverables. Responsible for data delivery in timely manner.

Detailed Job descriptions of lab personnel are found in training folders

4.2 ***Employee Screening, Orientation, and Training***.

All potential laboratory employees are screened and interviewed by human resources and technical staff prior to their hire. The pre-screen process includes a review of their qualifications including education, training and work experience to verify that they have adequate skills to perform the tasks of the job. Minimum qualifications for non-technical personnel require High School diploma (couriers also shall posses clean driving record), technical personnel must also demonstrate basic laboratory experience, such as balance and syringe use, aseptic practices, etc. College-level science coursework is favored.

Newly hired employees receive orientation training beginning the first day of employment by the Company. Orientation training consists of initial health and safety training and a detailed review of the personal protection policies, technical ethics training and data integrity procedures and quality assurance program training (including Company's goals, objectives, mission, and vision).

All technical staff receives training to develop and demonstrate proficiency for the methods they perform. New analysts work under supervision until the supervisory staff is satisfied that a thorough understanding of the method is apparent. Organics/Inorganics analysts are required to demonstrate method proficiency through a precision and accuracy study. Data from the study is compared to method acceptance limits. If the data is unacceptable, additional training is required. The analyst must also demonstrate the ability to produce acceptable data through the analysis of an independently prepared proficiency sample.

Proficiency is demonstrated annually. Data from initial and continuing proficiency demonstration is archived in the individual's training folder. In the instance where analyte can not be spiked in the clean matrix, such as TSS or pH, the results of an external Performance Evaluation (PE) sample may be used to document analyst's proficiency.

Minimum training required for administrative staff consists of laboratory safety and ethical conduct.

4.3 ***Training Documentation***. The QA Officer prepares a training file for every new employee. All information related to qualifications, experience, external training courses, and education are placed into the file. Verification documentation for

orientation, health & safety, quality assurance, and ethics training is also included in the file.

Additional training documentation is added to the file as it occurs. This includes data for initial and continuing demonstrations of proficiency, performance evaluation study data and notes and attendance lists from group training sessions.

The Quality Assurance Department maintains the employee training database. This database is a comprehensive inventory of training documentation for each individual employee. The database enables supervisors to obtain current status information on training data for individual employees on a job specific basis. It also enables the management staff to identify training documentation in need of completion.

Employee specific database records are created by human resources on the date of hire. Data base fields for job specific requirements such as SOP documentation of understanding and annual demonstration of analytical capability are automatically generated when the supervisor assigns a job responsibility. Employees acknowledge that their SOP responsibilities have been satisfied using a secure electronic process, which updates the database record. Reports are produced which summarize the qualifications of individual employees or departments.

5.0 SIGNATORY APPROVALS

Requirement: Procedures are required for establishing the traceability of data and documents. The procedure consists of a signature hierarchy, indicating levels of authorization for signature approvals of data and information within the organization. Signature authority is granted for approval of specific actions based on positional hierarchy within the organization and knowledge of the operation that requires signature approval. A log of signatures and initials of all employees is maintained for cross-referencing purposes.

5.1 *Signature Hierarchy*.

President/Chief Executive Officer. Authorization for contracts and binding agreements with outside parties. Approval of final reports, quality assurance policy, SOPs, project specific QAPs, data review and approval in lieu of technical managers. Contract signature authority resides with Company Officers only, which include the President/CEO, CFO and VP Administration.

Vice President, Operations/Laboratory Director. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. Technical policy.

Technical Director (on location): Approval of final reports and quality assurance policy in the absence of the Laboratory Director. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. Technical policy review. In the event of prolonged absence refer to list of approved deputies – sec 2.2.

Director, Quality Assurance. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers.

Quality Assurance Officer (on location). Approval of final reports and quality assurance policy in the absence of the Laboratory Director. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. In the event of prolonged absence refer to list or appointed deputies – see sec. 2.2.

Manager, Sample Management. Initiation of laboratory sample custody and acceptance of all samples. Approval of department policies and procedures. Department specific supplies purchase. Waste manifesting and disposal.

Project Manager, Client Services. QAP and sampling and analysis plan approval. Project specific contracts, pricing, and price modification agreements. Approval and acceptance of incoming work, Client services policy.

Supervisors, Technical Departments. Methodology and department specific QAPs. Data review and approval, department specific supplies purchase. Technical approval of SOPs.

Supervisors, Technical Departments. Data review approval, purchasing of expendable supplies.

5.2 ***Signature Requirements***. All laboratory activities related to sample custody and generation or release of data must be approved using either initials or signatures. The individual, who applies his signature or initial to an activity or document, is authorized to do so within the limits assigned to them by their supervisor. All signatures and initials must be applied in a readable format that can be cross-referenced to the signatures and initials log if necessary.

5.3 ***Signature and Initials Log***. The QA Officer maintains a signature and initials log. New Employee signatures and initials are appended to the log on the first day of employment. Signature of individuals no longer employed by the company are retained**.**

6.0 DOCUMENTATION and DOCUMENT CONTROL

Requirement: Document control policies have been established which specify that any document used as an information source or for recording analytical or quality control information must be managed using defined document control procedures. Accordingly, policies and procedures required for the control, protection, and storage of any information related to the production of analytical data and the operation of the quality system to assure its integrity and traceability have been established and implemented in the laboratory. The system contains sufficient controls for managing, archiving and reconstructing all process steps, which contributed to the generation of an analytical test result. Using this system, an audit trail for reported data can be produced, establishing complete traceability for the result.

6.1 Administrative Records. The Quality Assurance Officer manages Administrative (non-analytical) records. These records consist of electronic documents that are retained in a limited access electronic directory, which are released to the technical staff upon specific request.

Form Generation & Control. The Quality Assurance Officer approves all forms used as either stand-alone documents or in logbooks to ensure their traceability. Forms are generated as computer files only and maintained in a limited access master directory. Access to the electronic forms and applications is granted to QA Officer, Laboratory Manager and Technical Director(s) (local and regional). Approved forms must display the date of current revision and initials of person who revised the form. Modifications to existing forms are approved by QA, obsolete forms moved to archive directory and retained for minimum of five years.

New forms must include Accutest SE identification and appropriate spaces for signatures of approvals and dates. Further design specifications are the responsibility of the originating department.

Technical staff is required to complete all forms to the maximum extent possible. If information for a specific item is unavailable, the analyst is required to cross out the information block. The staff is also required to cross out the uncompleted portions of a logbook or logbook form if the day's analysis does not fill the entire page of the form.

Logbook Control. All laboratory logbooks are controlled documents that are comprised of approved forms used to document specific processes. Logbook control is maintained by QA staff.

New logs are numbered and issued to a specific individual who is assigned responsibility for the log. Supervisor performs periodical review of the logbooks. Old logs are returned to QA for entry into the document archive system where they are retained for minimum of five (5) years. Laboratory staff may hold a maximum of two consecutively dated logbooks of the same type in the laboratory, not including the most recently issued book to simplify review of recently completed analysis.

Controlled Documents. Key laboratory documents are designated for controlled document status to assure that identities of individuals receiving copies and the number of copies that have been distributed are known. Controlled status simplifies document updates and **retrieval** of outdated documents. Control is maintained through a document numbering procedure and document control logbook designating the individual receiving the controlled document. Document control is also maintained by pre-designating the numbers of official copies of documents that are placed into circulation within the laboratory.

Quality Systems Manual (QSM). All QSMs are assigned a number prior to distribution. The QSMs are distributed as controlled documents i.e. ones that will be collected back and replaced with next version (documents distributed to the Accutest Inc. staff). QSMs distributed to outside entities are considered tracked documents – since there is no possibility of collecting them back and ensuring that current revision is in use. These situation include bid submissions, client requests, etc. These copies are watermarked as "Uncontrolled Documents" The control/tracking number, date of distribution, and identity of the individual receiving the document are recorded in the document control spreadsheet. QA staff maintains tracking spreadsheet. The numbering system is continuous.

Standard Operating Procedures (SOPs). SOPs are maintained by pre-designating the numbers of official copies of documents that are placed into circulation within the laboratory. Official documents are printed and placed into the appropriate laboratory section as follows:

Sample Management: One copy for the sample receiving file
Bottle preparation area – One copy for shipping area
Organics Laboratories: One for the affected laboratory area.
Inorganics Laboratories: One for the affected laboratory area.

The original, signed copy of the SOP is maintained in the master SOP binder by the QA staff.

Documents are controlled using an "Official Copy" stamp in red ink. Additional copies could be issued to individuals for training purposes. Distribution is documented on SOP cover page. Superceded copies collection is conducted accordingly to cover page distribution list.

SOPs distributed to clients as part of bid submission, pre-audit evaluation, etc. are watermarked as "Proprietary Information".

Quick reference cards: These documents are compiled for lab staff convenience and are based on current SOP revision and/or recent regulatory updates. These one- or two-sided documents are footnoted with reference to SOP/regulatory standard, stamped with "Official Copy" stamp in red ink and laminated for durability. *Use of these quick references does not substitute reading and acknowledging the parent SOP.*

Operators' Manuals are considered controlled documents and stored in appropriate departments.

6.2 ***Technical Records***. All records related to the analysis of samples and the production of analytical results are archived in secure document storage or on electronic media and contain sufficient detail to produce an audit trail, which re-creates the analytical result. These records include information related to the original client request, bottle order, sample login and custody, storage, sample preparation, analysis, data review and data reporting.

Records that can not be maintained on electronic media are considered irretrievable records, segregated into separate secured storage and access controlled with access log maintained by QA Staff. Examples of such records are employee training files, obsolete SOPs and acknowledgement form originals, training files, logbooks, etc.

Each department involved in this process maintains controlled documents, which enable them to maintain records of critical information relevant to their department's process.

6.3 ***Quality Assurance Directory.*** All Quality Assurance documentation and quality control limit data is stored in a restricted QA directory on the network server. The directory has been designated as read only. The QA staff, technical director and the laboratory manager have write capability in this directory. Information on this directory is backed-up daily.

This directory contains all current and archived Quality System Manuals, SOPs, control limits, MDL studies, precision and accuracy data, internal and external audit reports, official forms, Health and Safety materials, PT scores, State Certifications and metrics calibration information.

6.4 ***Analytical Records***. All data related to the analysis of field samples are retained as either paper or electronic records that can be retrieved to compile a traceable audit trail for any reported result. All information is linked to the client job and sample number, which serves as a reference for all sample related information tracking.

Critical times in the life of the sample from collection through analysis to disposal are documented. This includes date and time of collection, receipt by the laboratory, preparation times and dates, analysis times and dates and data reporting information. Analysis times are calculated in hours for methods where holding time is specified in hours (≤72 hours).

Sample preparation information is recorded in a separate controlled logbook or on controlled forms in three-ring binder. It includes sample identification numbers, types of analysis, preparation and cleanup methods, sample weights and volumes, reagent lot numbers and volumes and any other information pertinent to the preparation procedure.

Information related to the identification of the instrument used for analysis is permanently attached to the electronic record. The record includes an electronic data file that indicates all instrument conditions employed for the analysis, including the type of analysis conducted. The analyst's identification is electronically attached to the record. The instrument tuning and calibration data is electronically linked to the sample or linked though paper logs, which were used in the documentation of the analysis. Quality control and performance criteria are permanently linked to the paper archive or electronic file.

Paper records for the identity, receipt, preparation and evaluation of all standards and reagents used in the analysis are documented in prepared records and maintained in controlled documents or files. Lot number information linking these materials to the analysis performed is recorded in the logbooks associated with the samples in which they were used.

Manual calculations or peak integrations that were performed during the data review are retained as paper or electronically generated PDF documents and included as part of the electronic archive. Signatures for data review are retained on paper or as electronic stamps on PDF versions of the paper record for the permanent electronic file.

6.5 ***Confidential Business Information (CBI).*** Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between Accutest and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as Adobe Acrobat .PDF format only.

6.6 ***Software Change Documentation & Control.*** Changes to software are documented as text within the code of the program undergoing change. Documentation includes a description of the change, reason for change and the date the change was placed into effect. Documentation indicating the adequacy of the change is prepared following the evaluation by the user who requested the change.

6.7 ***Report and Data Archiving***. Accutest Laboratories maintains electronic image file copies of original reports in archive for a minimum period of five (5) years. After five years, the files are automatically discarded unless contractual arrangements exist which dictate different requirements. Client specific data retention practices are employed for government organizations such as the Department of Defense Agencies and MA DEP that require a retention period of ten (10) years, as well as commercial clients upon contractual requirements agreement.

Complete date and time stamped client reports are generated from LIMS using the source documents archived on Document server. These source documents are maintained on document server and backed up to primary and clone tapes. Accutest

archives the original report (organized by job number) and the organic and inorganic support data. Organic support data is archived according to instrument batch numbers. All organics data is backed up to the tape or archive drive via Networker Backup software and/or AccuBack backup software. Data from the archive drive is then written to tape at periodic intervals.

Wet chemistry support data is archived by analytical batch (GN…). Metals support data is archived by instrument batch (MA…). Metals digestion data is archived as digestion logbooks.

The reports generation group electronically scans completed reports and stores them by job number on the document server. The document server is backed up daily to a digital tape. Copies of these files remain active on the document server for easy review access. The digital tapes remain in secure storage for the remainder of the archive period.

6.8 ***Training***. Ongoing training ensures competence of all relevant personnel. At the minimum personnel should possess knowledge of the technology used in the testing, general requirements expressed in legislature and industry standards, and understand the significance of deviations with regard to approved procedures. The company maintains a training record for all employees that documents that they have received instruction on administrative and technical tasks that are required for the job they perform. Training records for individuals employed by the company are retained for a period of five years following their termination of employment.

Training File Origination. The Quality Assurance Officer (QAO) initiates training files. Quality Assurance officer retains the responsibility for the maintenance and tracking of all training related documentation in the file. The file is begun on the first day of employment. Information required for the file includes a copy of the individual's most current resume, detailing work experience and a copy of any college diplomas or transcript(s). Information added on the first day includes documentation of health and safety training and a signed Ethics and Data Integrity agreement. These two constitute minimal necessary training for Project Management and Administrative staff. Training documentation, training requirements, analyst proficiency information and other training related support documentation is tracked using a customized database application. Database extracts provide an itemized listing of specific training requirements by job function. Training status summaries for individual analysts portray dates of completion for job specific training requirements.

Technical Training. The supervisor of each new employee is responsible for developing a training plan for each new employee. The supervisor updates the outline, adding signatures and dates as training elements are completed at regular frequency. Supporting documentation, such as precision and accuracy studies, which demonstrate analyst capability for a specific test, are added as completed. When analyte can not be spiked, such as pH or TSS, external PE sample is purchased and analyzed. Where no external PE sample is available, sample duplicates must be successfully analyzed. Method review records are retained where analysis of duplicates is not possible. Employees and supervisors verify documentation of

understanding (DOU) for all assigned standard operating procedures in the training database. Certificates or diplomas for any off-site training are added to the file.

7.0 REFERENCE STANDARD TRACEABILITY

Requirement: Documented procedures, which establish traceability between any measured value and a national reference standard, must be in place in the laboratory. All metric measurements must be traceable to NIST reference weights or thermometers that are calibrated on a regular schedule. All chemicals used for calibration of a quantitative process must be traceable to an NIST reference that is documented by the vendor using a certificate of traceability. The laboratory maintains a documentation system that establishes the traceability links. The procedures for verifying and documenting traceability must be documented in standard operating procedures.

7.1 ***Traceability of Metric Measurements - Thermometers***. Accutest uses NIST-traceable thermometers to calibrate commercially purchased working laboratory thermometers prior to their use in the laboratory and annually thereafter for liquid in glass thermometers or quarterly for electronic temperature measuring devices. If necessary, these working thermometers are assigned correction factors that are determined during their calibration using an NIST-traceable thermometer as the standard. The correction factor is documented in a thermometer log and on a tag attached to the working thermometer. Both original observation and corrected measurement are recorded in the temperature log. The NIST-traceable reference thermometer is checked for accuracy by an outside vendor minimum every five (5) years following the specifications for NIST-traceable thermometer calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005. Currently the NIST thermometer is verified by outside vendor on triennial basis due to contract-specific requirements. Calibration log and Certificate(s) of calibration are maintained on file with QAO.

7.2 ***Traceability of Metric Measurements – Calibration Weights***. Accutest uses calibrated weights, which are traceable to NIST standard weights to calibrate all balances used in the laboratory. Balances must be calibrated to specific tolerances within the intended use range of the balance. Calibration checks are required on each day of use. If the tolerance criteria are not achieved, corrective action specified in the balance calibration SOP must be applied before the balance can be used for laboratory measurements. All weights are recalibrated by outside vendor every five years following the specifications for weight calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005. Certificate(s) of calibration are maintained on file with QAO. Balances are inspected and maintained by professional service technicians annually. Certificate(s) of inspection are maintained with QAO.

7.3 ***Traceability of Chemical Standards and Reagents***. All chemicals and reagents, with the exception of bulk dry Na2SO4 and solvents purchased as reference standards for use in method calibration must establish traceability to NIST referenced material through a traceability certificate (Certificate of Analysis, CoA). Process links are

established that enable a calibration standard solution to be traced to its NIST reference certificate. Solvents, acids and other supplies are being tested to verify their suitability for the analytical process.

7.4 ***Assignment Of Reagent and Standard Expiration Dates.*** Expiration date information for all purchased standards and reagents is provided to Accutest with all prepared standard solutions and unstable reagents as a condition of purchase. Neat materials and inorganic reagents are not required to be purchased with expiration dates. Certified prepared solutions are labeled with the expiration date provided by the manufacturer. In-house prepared solutions are assigned expiration dates that are consistent with the method that employs their use unless documented experience indicates that an alternate date can be applied. If alternate expiration dates are employed, their use is documented in the method SOP. Expiration dates for prepared inorganic reagents, which have not exhibited instability, are established at two years form the date of preparation for tracking purposes. All containers shall be labeled with the date of preparation and expiration date clearly indicated.

The earliest expiration date is always the limiting date for assigning expiration dates to prepared solutions. Expiration dates that are later than the expiration date of any derivative solution or material are prohibited.

7.5 ***Documentation of Traceability***. Traceability information is documented in individual logbooks designated for the measurement process in use. The QA Officer maintains calibration documentation for metric references in pertinent folders and logbooks.

Balance calibration verification is documented in logbooks that are assigned to each balance. The individual conducting the verification is required to initial and date all calibration activities. Any defects that occur during verification are also documented along with the corrective action applied and a demonstration of return to control. Annual service and calibration reports and certificates retained on file with QA staff.

Temperature control is documented in logbooks assigned to the equipment being monitored. A verified (see 7.1) thermometer is assigned to each individual item. Measurements are recorded along with date and initials of the individual conducting the measurement on a daily or as used basis. Corrective action, if required, is also documented including the demonstration of return to control.

Initial traceability of chemical standards and reagents is documented via a vendor-supplied certificate (see also 7.3) that includes lot number and expiration date information. Solutions prepared using the vendor supplied chemical standard are documented in logbooks assigned to specific analytical processes. Alternatively, documentation may be entered into the electronic standards and reagent tracking log The documentation includes links to the vendors lot number, an internal lot number, dates of preparation, and the preparer's initials. Standards received without certificate of analysis can not be used for calibration or calibration verification and are rejected.

Supervisors conduct regular reviews of logbooks, which are verified using a word rev'd", signature and date. QA Staff monitors the process and documents it in the same manner.

8.0 TEST PROCEDURES, METHOD REFERENCES, AND REGULATORY PROGRAMS

Requirements: The laboratory must use client specified or regulatory agency approved methods for the analysis of environmental samples. The laboratory maintains a list of active methods, which specifies the type of analysis performed, and cross-references the methods to applicable environmental regulation. Routine procedures used by the laboratory for the execution of a method must be documented in a standard operating procedure. Method performance and sensitivity must be demonstrated annually where required. Defined procedures for the use of method sensitivity for data reporting purposes must be established by the Director of Quality Assurance and used consistently for all data reporting purposes.

8.1 ***Method Selection.*** Accutest employs methods for environmental sample analysis that are consistent with the client's application, which are appropriate and applicable to the project objectives. Accutest informs the client if the method proposed is inappropriate or outdated and suggests alternative approaches.

Accutest employs documented, validated regulatory methods in the absence of a client specification and informs the client of the method selected. These methods are available to the client and other parties as determined by the client. Documented and validated in-house methods may be applied if they are appropriate to the project. The client is informed of the method selection.

8.2 ***Method Validation***. Standard methods from regulatory sources are primarily used for all analysis. Standard methods do not require validation by the laboratory. Non-standard, in-house methods are validated prior to use. Validation is also performed for standard methods applied outside their intended scope of use. Validation is dependent upon the method application and may include analysis of quality control samples to develop precision and accuracy information for the intended use. A final method validation report is generated, which includes all data in the validation study. A statement of adequacy and/or equivalency is included in the report. A copy of the report is archived in the quality assurance directory of the company server.

Non-standard methods are validated prior to use. This includes the validation of modified standard methods to demonstrate comparability with existing methods. Demonstrations and validations are performed and documented prior to incorporating technological enhancements and non-standard methods into existing laboratory methods used for general applications. The demonstration includes method specific requirements for assuring that significant performance differences do not occur when the enhancement is incorporated into the method. Validation is dependent upon method application and may include the analysis of quality control samples to develop precision and accuracy information for intended use.

The study procedures and specifications for demonstrating validation include comparable method sensitivity, calibration response, method precision, method accuracy and field sample consistency for several classes of analytical methods are

detailed in this document. These procedures and specifications may vary depending upon the method and the modification.

8.3 ***Standard Operating Procedures***. Standard operating procedures (SOP) are prepared for routine methods executed by the laboratory and processes related to sample or data handling. The procedures describe the process steps in sufficient detail to enable an individual, who is unfamiliar with the procedure to execute it successfully. SOPs are reviewed annually and edited if necessary. SOPs can be edited on a more frequent basis if systematic errors dictate a need for process change or the originating regulatory agency promulgates a new version of the method. Procedural modifications are indicated using a revision number. SOPs are available for client review at the Accutest facility upon request.

8.4 ***Method Detection Limit Determination and verification***. Annual method detection limit (MDL) studies are performed as appropriate for routine methods used in the laboratory. MDL studies are also performed when there is a change to the method that affects how the method is performed or when an instrumentation change that impacts sensitivity occurs. The procedure used for determining MDLs is described in 40 CFR, Part 136, Appendix B. Studies are performed for each method on water, soil and air matrices for every instrument that is used to perform the method. MDLs are established at the instrument level. The highest MDL of the pooled instrument data is used to establish a laboratory MDL. MDLs are experimentally verified through the analysis of spiked quality control samples at 2-3 times the concentration of the experimental MDL, or 1-4 times for multicomponent methods. The verification is performed on every instrument used to perform the analysis. The quality assurance staff manages the annual MDL determination process and is responsible for retaining MDL data on file. Approved MDLs are appended to the LIMS and used for data reporting purposes. MDL values are used as DL values for DOD projects and verification spiking concentrations are listed ad LOD values.

Methods certified under DOD ELAP requirements must undergo verification procedure on quarterly basis – see DOD QSM 4.2, Gray Box D-13.

8.5 ***Method Reporting Limit.*** The method reporting limit is established at the lowest concentration calibration standard in the calibration curve. The low calibration standard is selected by department managers as the lowest concentration standard that can be used while continuing to meet the calibration linearity criteria of the method being used. The validity of the Method Reporting Limits is confirmed via analysis of a spiked quality control sample at 1 – 2x Method reporting limit concentration. RL values are referred to as LOQ for DOD projects.

By definition, detected analytes at concentrations below the low calibration standard cannot be accurately quantitated and must be qualified accordingly.

Methods certified under DOD ELAP requirements must undergo verification procedure on quarterly basis – see DOD QSM 4.2, Gray Box D-14.

8.6 ***Reporting of Quantitative Data.*** Analytical data for all methods is reported without qualification to the reporting limit established for each method. Data may be reported to MDL depending upon the client's requirements provided that all qualitative identification criteria for the parameter have been satisfied. All parameters reported at concentrations between the reporting limit and MDL are qualified as an estimated concentration.

Measured concentrations of detected analytes that exceed the upper limit of the calibration range are either diluted into the range and reanalyzed or qualified as an estimated value. The only exception to this applies to ICP and ICP/MS analysis, which can be reported to the upper limit of the experimentally determined linear range without qualification.

8.7 ***Estimated Uncertainty.*** A statement of the estimated uncertainty of an analytical measurement accompanies the test result when required. Estimated uncertainty is derived from the performance limits established for spiked samples of similar matrices. The degree of uncertainty is derived from the negative or positive bias for spiked samples accompanying a specific parameter. When the uncertainty estimate is applied to a measured value, the possible quantitative range for that specific parameter at that measured concentration is defined. Well recognized regulatory methods that specify values for the major sources of uncertainty and specify the data reporting format do not require a further estimate of uncertainty.

8.8 ***Precision and Accuracy Studies***. Annual precision and accuracy (P&A) studies, which demonstrate the laboratories ability to generate acceptable date, are performed for all routine methods used in the laboratory. The procedure used for generating P&A data is referenced in the majority of the regulatory methodology in use. The procedure requires quadruplicate analysis of a sample spiked with target analytes at a concentration in the working range of the method. This data may be compiled from a series of existing blank spikes or laboratory control samples. Accuracy (percent recovery) of the replicate analysis is averaged and compared to established method performance limits. Values within method limits indicate an acceptable performance demonstration. (See also Sec 4, Training, Demonstration of capability)

8.9 ***Method Sources, References and Update Mechanism.*** The Quality Assurance Staff maintains a list of active methods used for the analysis of samples. This list includes valid method references such as EPA, American Society of Testing and Materials (ASTM) or Standard Methods designations and the current version and version date.

Updated versions of approved reference methodology are placed into use as changes occur. The Quality Assurance Director informs operations management of changes in method versions as they occur. The operations management staff selects an implementation date. The operations staff is responsible for completing all method requirements prior to the implementation date. This includes modification to SOPs, completion of MDL and precision and accuracy studies and staff training. Documentation of these activities is provided to the QA staff who retains this information on file. The updated method is placed into service on the implementation date and the old version is de-activated.

Multiple versions of selected methods may remain in use to satisfy client specific needs. In these situations, the default method version becomes the most recent version. Client specific needs are communicated to the laboratory staff using method specific analytical codes method, which clearly depict the version to be used. The old method version is maintained as an active method until the specified client no longer requires the use of the older version.

Accutest will not use methodology that represents significant departures from the reference method unless specifically directed by the client. In cases where clients direct the laboratory to use a method modification that represents a significant departure from the reference method, the request will be documented in the project file. The LQSM lists active methods used for the analysis of samples in Table 8.1. This list includes valid method references from sources such as USEPA, ASTM or Standard Methods designations and the current version and version date.

8.10 ***Analytical Capabilities***. Appendix II provides a detailed listing of the methodology employed for the analysis of test samples.

9.0 SAMPLE MANAGEMENT, LOGIN, CUSTODY, STORAGE AND DISPOSAL

Requirement: A system to ensure that client supplied product is adequately evaluated, acknowledged, and secured upon delivery to the laboratory must be practiced by the laboratory. The system must assure that chain of custody is maintained and that sample receipt conditions and preservation status are documented and communicated to the client and internal staff. The login procedure must assign, document, and map the specifications for the analysis of each unique sample to assure that the requested analysis is performed on the correct sample and enables the sample to be tracked throughout the laboratory analytical cycle. The system must include procedures for reconciling defects in sample condition or client provided data, which occur at sample arrival. The system must specify the procedures for proper sample storage, transfer to the laboratory, and disposal after analysis. The system must be documented in a standard operating procedure.

9.1 ***Order Receipt and Entry***. New orders are initiated and processed by the client services group (See Chapter 14, Procedures for Executing Client Specifications). The new order procedure includes mechanisms for providing sampling containers to clients. These containers must meet the size, cleanliness, and preservation specifications for the analysis to be performed.

For new orders, the project manager prepares a bottle request form, which is submitted to sample management department. This form provides critical project details to the sample management staff, which are used to prepare and assemble the sample bottles for shipment to the client prior to sampling.

The bottle order is assembled using bottles that meet USEPA specifications for contaminant-free sample containers. Accutest-SE checks all sample containers for cleanliness. Data are reviewed by both the analyst and sample management technician. Results of bottle analyses are retained for minimum of 5 years.

All preservative solutions are prepared in the laboratory and are checked to assure that they are free of contamination from analytes of interest before being released for use. Sample management department retains a copy of the documentation of in-house contamination checks.

Reagent water for trip and field blanks is poured into appropriately labeled containers. Sample bottleware is labeled with durable labels printed on waterproof printing medium with indelible laser or heat transfer printer ink. All bottles are packed into ice chests with blank chain of custody forms and the original bottle order form. Completed bottle orders are delivered to clients using Accutest couriers or commercial carriers for use in field sample collection.

9.2 ***Sample Receipt and Custody***. Samples are delivered to the laboratory using a variety of mechanisms including Accutest couriers, commercial shippers, and client self-delivery. Documented procedures are followed for arriving samples to assure that

custody and integrity are maintained and that handling and preservation requirements are documented and continued.

Sample custody documentation is initiated when the individual collecting the sample collects field samples. Custody documentation includes all information necessary to provide an unambiguous record of sample collection, sample identification, and sample collection chronology. Initial custody documentation employs either Accutest or client generated custody forms.

Accutest generates a Sample Receipt Confirmation form in situations where the individuals who collected the sample did not generate custody documentation in the field. Accutest SE Project Manager then contacts the client for the CoC information to be faxed or e-mailed from the client to the lab.

Accutest defines sample custody as follows:

- The sample is in the actual custody or possession of the assigned responsible person,
- The sample is in a secure area.

The Accutest facility is defined as a secure facility. Perimeter security has been established, which limits access to authorized individuals only. Visitors enter the facility through the building lobby and must register with the receptionist prior to entering controlled areas. While in the facility, visitors must be accompanied by their hosts at all times. After hours, building access is controlled using a computerized pass-key reader system. This system limits building access to individuals with a pre-assigned authorization status. After hours visitors are not authorized to be in the building. Clients delivering samples after hours must make advanced arrangements through client services and sample management to assure that staff is available to take delivery and maintain custody.

Upon arrival at Accutest, the sample custodian reviews the chain of custody and generates Sample Receipt Confirmation form for the samples received to verify that the information on the form corresponds with the samples delivered. This includes verification that all listed samples are present and properly labeled, checks to verify that samples were transported and received at the required temperature, verification that the sample was received in proper containers, verification that sufficient volume is available to conduct the requested analysis, and a check of individual sample containers to verify test specific preservation requirements including the absence of headspace for volatile compound analysis.

9.3 Sample conditions and other observations are documented on the Sample Receipt Confirmation form by the sample custodian prior to completing acceptance of custody. The sample custodian accepts sample custody upon verification that the custody document is correct. Discrepancies or non-compliant situations are documented, flagged and communicated to the Accutest project manager, who contacts the client

for resolution. The resolution is documented and communicated to sample management for execution.

9.4 ***Laboratory preservation of Improperly preserved field samples.*** Accutest extends every effort to preserve samples which were received without proper field preservation.

Field/Equipment negative controls also receive the same amount of preservation as incorrectly preserved samples, and record made in the preservation logbook.

9.5 ***Sample Tracking Via Status Change.*** An automated, electronic LIMS procedure records sample exchange transactions between departments and changes in analytical status. This system tracks all preparation, analytical, and data reporting procedures to which a sample is subjected while in the possession of the laboratory. Each individual receiving samples must acknowledge the change in custody and operational status in the LIMS. This step is required to maintain an accurate electronic record of sample status, dates of analytical activity, and custody throughout the laboratory.

Sample tracking is initiated at login where all chronological information related to sample collection dates and holding times are entered into the LIMS. This information is entered on an individual sample basis

9.6 ***Sample Acceptance Policy***. Incoming samples must satisfy Accutest's sample acceptance criteria before being logged into the system. Sample acceptance is based on the premise that clients have exercised proper protocols for sample collection. This includes sufficient volume, proper chemical preservation, temperature preservation, sample container sealing and labeling, and appropriate shipping container packing.

The sample management staff will make every attempt to preserve improperly preserved samples upon arrival. However, if preservation is not possible, the samples may be refused unless the client authorizes analysis. No samples will be accepted if holding times have been exceeded or will be exceeded before analysis can take place unless the client authorizes analysis.

Sample acceptance criteria include proper custody and sample labeling documentation. Proper custody documentation includes an entry for all physical samples delivered to the laboratory with an identification code that matches the sample bottle and a date and signature of the individual who collected the sample and delivered them to the laboratory. Labeling is done using durable waterproof labels printed with indelible heat-transfer ink.

Accutest reserves the right to refuse any sample which in its sole and absolute discretion and judgement is hazardous, toxic and poses or may pose a health, safety or environmental risk during handling or processing. The company will not accept samples for analysis using methodology that is not performed by the laboratory or for methods that lab does not hold valid accreditation unless arrangements have been made to have the analysis conducted by a qualified subcontractor.

9.7 ***Assignment of Unique Sample Identification Codes***. Unique identification codes must be assigned to each sample bottle to assure traceability and unambiguously identify the tests to be performed in the laboratory.

The sample identification coding process begins with the assignment of a unique alphanumeric job number. A job is defined as a group of samples received on the same day, from a specific client pertaining to a specific project. A job may consist of groups of samples received over multi-day period. The first character of the job number is an alpha-character that identifies the laboratory facility. The next characters are numeric and sequence by one number with each new job.

Unique sample numbers are assigned to each bottle collected as a discrete entity from a designated sample point. This number begins with the job number and incorporates a second series of numbers beginning at one and continuing chronologically for each point of collection. The test to be performed is clearly identified on the bottle label.

Alpha suffixes may be added to the sample number to identify special designations such as subcontracted tests, in-house QC checks, or re-logs. Multiple sample bottles for a specific analysis are labeled Bottle 1, Bottle 2, etc.

9.8 ***Subcontracted Analysis***. Subcontract laboratories are employed to perform analysis not performed by Accutest. The quality assurance staff evaluates subcontract laboratories to assure their quality processes meet the standards of the environmental laboratory industry prior to engagement. Throughout the subcontract process, Accutest follows established procedures to assure that sample custody is maintained and the data produced by the subcontractor meets established quality criteria.

Accutest network laboratories are considered primary subcontractors.

*Subcontracting Procedure***.** Subcontracting procedures are initiated through several mechanisms, which originate with sample management. Samples for analysis by a subcontractor are logged into the Accutest system using regular login procedures. If subcontract parameters are part of the project or sample management has received subcontracting instructions for a specific project, a copy of the chain of custody is given to the appropriate project manager with the subcontracted parameters highlighted. This procedure triggers the subcontract process at the project management level. The Sample Management supervisor contacts an approved subcontractor to place the subcontract order. Subcontract chain of custody is processed in Sample Management Department and copy is filed with the original CoC. Sample management signs the subcontract chain of custody and ships the sample(s) to the subcontractor. The subcontract COC is filed with the original COC and the request for subcontract. Copies are distributed to the login department, the project manager, and sample management.

Client is verbally notified by Project Manager of the requirement to subcontract to the outside laboratory as soon as need Is identified by the Accutest staff. Client notification

must be verified in writing, i.e. by e-mail. Client notification may take place during the initial project set-up, or at the time of sample receipt and login.

Subcontractor data packages are reviewed by the QA Staff to assess completeness and quality compliance. If completeness defects are detected, the subcontractor is asked to immediately upgrade the data package. If data quality defects are detected, the package is forwarded to the QA staff for further review. The QA staff will pursue a corrective action solution before releasing data to the client.

Approved subcontract data is entered into the laboratory information management system (LIMS) if possible and incorporated into the final report. All subcontract data is footnoted to provide the client with a clear indication of its source. Copies of original subcontract data are always included in the data report whether in hardcopy or PDF file, depending on the data submission requirements.

*Subcontract Laboratory Evaluation***.** The QA staff evaluates subcontract laboratories prior to engagement. As a minimum, the subcontract laboratory must provide Accutest with proof of a valid certification to perform the requested analysis for the venue where they were collected, QC criteria summary (LOD/LOQ, LCS, MS/MSD, %RPD, etc.), copy of the most recent regulatory agency audit report, and a copy of the laboratory's Summary of Qualifications (SOQ). Other beneficial materials are QSM, copies of SOPs used for the subcontracted analysis, a copy of the most recent performance evaluation study for the subcontracted parameter, and copies of the most recent third party accreditor's audit report.

Certification verification must be submitted to Accutest annually. If possible, the QA staff may conduct a site visit to the laboratory to inspect the quality system. Accutest Laboratories Southeast assumes the responsibility for the performance of all subcontractors who have successfully demonstrated their qualifications. When selecting a subcontractor for analysis not performed by Accutest, assure qualifications of the subcontractor through local QA officer.

Qualification process of a subcontract laboratory may be bypassed if the primary client directs Accutest to employ a specific subcontractor

Subcontract Laboratory Database. Accutest Laboratories Inc. maintains centralized database of preferred contractors in order to optimize sample handling and data submission process, as well as obtain competitive priced services of uniform quality throughout the network. Individual Accutest laboratories are assigned "Center of Expertise" status according to unique capabilities.

9.9 ***Sample Storage***. Following sample custody transfer, samples are assigned to various refrigerated storage areas by the sample management staff depending upon the test to be performed and the matrix of the samples. The location (refrigerator and shelf) of each sample is entered into sample location database on the line corresponding to each sample number. Samples remain in storage until the laboratory technician retrieves them into the laboratory for analysis.

Samples for volatile organics analysis are placed in storage in designated refrigerators by the sample management staff and immediately transferred to the organics group control. Sample custody is transferred to the VOC department staff. These samples are segregated according to matrix to limit opportunities for cross contamination to occur.

Organics staff is authorized to retrieve samples from these storage areas for analysis. When analysis is complete, the samples are placed back into storage.

9.10 ***Sample Login***. Following sample custody transfer to the laboratory, the documentation that describes the clients analytical requirements are delivered to the sample login group for coding and entry to the Laboratory Information management System (LIMS). This process translates all information related to collection time, turnaround time, sample analysis, and deliverables into a code which enables client requirements to be electronically distributed to the various departments within the laboratory for scheduling and execution.

The technical staff is alerted to client or project specific requirements through the use of a unique project code that is electronically attached to the job during login. The unique project code directs the technical staff to controlled specifications documents detailing the unique requirements.

9.11 ***Sample Retrieval for Analysis***. It is a responsibility of individual analyst to retrieve samples for analysis. Sample Management employs a program to facilitate sample placement and retrieval. Sample is traced around the laboratory using Status feature of LIMS.

After sample analysis has been completed, the analyst places the sample back into the storage and updates sample status.

9.12 ***Sample Disposal***. Accutest retains all samples under proper storage for a minimum of 30 days following completion of the analysis report. Longer storage periods are accommodated on a client specific basis if required. Samples may also be returned to the client for disposal.

Accutest disposes of all laboratory wastes following the requirements of the Resource Conservation and Recovery Act (RCRA). The Company has obtained and maintains a waste generator identification number, FLR00001263309002 (FLR designates State of Florida).

Sample management generates a sample disposal dump sheet from the LIMS tracking system each week, which lists all samples whose holding period has expired. Data from each sample is compared to the hazardous waste criteria established by the Florida Department of Environmental Protection (FDEP).

Samples containing constituents at concentrations above the criteria are labeled as hazardous and segregated into the following waste categories for disposal as follows:

Chlorinated Waste (Closed Top Steel Drum)- Methylene Chloride

Non-Chlorinated Waste (Closed Top Steel Drum)- Hexane, Methanol, and mixed solvents

Sodium Sulfate/Used Charcoal (Open Top Steel Drum)- Charcoal and paper filters used in the filtering of samples.

Hazardous Flammable Vials (Open Top Polypropylene Drum)- Methylene Chloride, Hexane.

Hazardous Aqueous waste (Closed Top Polypropylene Drum)- High Odor Samples, Lachat Waste.

Non Hazardous Soil (Open Top Steel Drum)- Soils.

Hazardous Solid Waste- (Open Top Steel Drum).

Non-Aqueous/Oil Samples- (Closed Top Steel Drum)

Difference between Open and Closed type of drums is whether it is possible to remove entire lid or just threaded stopper. Drums are closed at all times while in storage.

Non-hazardous aqueous samples are neutralized and collected in HDPP 500 Gal holding tank to be removed by waste company.

Non-hazardous solids are drummed and disposed of by contract waste company. Sample bottles are disposed of as recyclable waste in order to crush the bottles and destroy the labels. VOC vials are crushed on site using PRODEVA glass crusher. Supernatant liquid is siphoned off into the HDPP holding tank and solid residue drummed separately.

Laboratory wastes are collected by waste stream in designated areas throughout the laboratory. Waste streams are consolidated twice a week by the waste custodian and transferred to stream specific drums for disposal through a permitted waste management contractor. Filled, consolidated drums are tested for hazardous characteristics and scheduled for removal from the facility for appropriate disposal based on the laboratory data.

10.0 LABORATORY INSTRUMENTATION AND MEASUREMENT STANDARDS

Requirement: Procedures, which assure that instrumentation is performing to a pre-determined operational standard prior to the analysis of any samples, must be established by the laboratory. In general, these procedures will follow the regulatory agency requirements established in promulgated methodology. The instrumentation selected to perform specified analysis is capable of providing the method-specified uncertainty and sufficient sensitivity of measurement needed. These procedures must be documented and incorporated into the standard operating procedures for the method being executed. ALSE Equipment List attached as Appendix III.

10.1 ***Mass Tuning – Mass Spectrometers***. The mass spectrometer tune and sensitivity must be monitored to assure that the instrument is assigning masses and mass abundances correctly and that the instrument has sufficient sensitivity to detect compounds at low concentrations. This is accomplished by analyzing a specific mass tuning compound at a fixed concentration. If the sensitivity is insufficient to detect the tuning compound, corrective action must be performed prior to the analysis of standards or samples. If the mass assignments or mass abundances do not meet criteria, corrective action must be performed prior to the analysis of standards or samples.

10.2 ***Wavelength Verification – Spectrophotometers***. Spectrophotometer detectors are checked on a regular schedule to verify proper response to the wavelength of light needed for the test in use. If the detector response does not meet specifications, corrective action (detector adjustment or replacement) is performed prior to the analysis of standards or samples.

10.3 ***Inter-element Interference Checks (Metals)***. Inductively Coupled Plasma Emission Spectrophotometers (ICP) are subject to a variety of spectral interferences, which can be minimized or eliminated by applying interfering element correction factors and background correction points. Interfering element correction factors are checked on a specified frequency through the analysis of check samples containing high levels of interfering elements. Analysis of single element interferent solutions is also conducted at a specified frequency.

If the check indicates that the method criteria has not been achieved for any element in the check standard, the analysis is halted and data from the affected samples are not reported. Sample analysis is resumed after corrective action has been performed and the correction factors have been re-calculated.

New interfering element correction factors are calculated and applied whenever the checks indicate that the correction factors are no longer meeting criteria. At a minimum, correction factors are replaced once a year.

10.4 ***Calibration and Calibration Verification***. Many tests require calibration using a series of reference standards to establish the concentration range for performing quantitative analysis. Method specific procedures for calibration are followed prior to any sample analysis.

Calibration is performed using a linear or quadratic regression calculation or calibration factors calculated from the curve. The calibration must meet method specific criteria for linearity or precision. If the criteria are not achieved, corrective action (instrument maintenance or re-calibration) is performed. The instrument must be successfully calibrated before analysis of samples can be conducted.

Initial calibration for metals analysis performed using inductively coupled plasma (ICP) employs the use of two standards and a calibration blank to establish linearity. The calibration blank contains all reagents that are placed into the calibration standard with the exception of the target elements. Valid calibration blanks must not contain any target elements.

Initial calibrations must be initially verified using a single concentration calibration standard from a second source (i.e. separate lot or different provider). The continuing validity of an existing calibration must be regularly verified using a single concentration calibration standard. The response to the standard must meet pre-established criteria that indicate the initial calibration curve remains valid. If the criteria are not achieved corrective action (re-calibration) is performed before any additional samples may be analyzed.

10.5 ***Linear Range Verification and Calibration*** Linear range verification is performed for all ICP instrumentation and select General Chemistry methods. The regulatory program or analytical method specifies the verification frequency. A series of calibration standards are analyzed over a broad concentration range. The data from these analyses are used to determine the valid analytical range for the instrument.

Some methods or analytical programs require a low concentration calibration check to verify that instrument is sufficient to detect target elements at the reporting limit. The analytical method or regulatory program defines the criteria used to evaluate the low concentration calibration check. If the low calibration check fails criteria, corrective action is performed and verified through reanalysis of the low concentration calibration check before continuing with the field sample analysis.

In accordance with TNI standards minimum number of calibration points in the absence of method-specific requirements is two calibration points and a blank.

10.6 ***Retention Time Verification (GC/HPLC/IC)***. Chromatographic retention time windows are developed for all analysis performed using gas chromatographs with conventional detectors. An initial experimental study is performed, which establishes the width of the retention window for each compound. The retention time range of the window defines the time ranges for elution of specified target analytes on the primary and

confirmation columns. Retention time windows are established upon initial calibration, applying the retention time range from the initial study to each target compound. Retention times are regularly confirmed through the analysis of an authentic standard during calibration verification. If the target analytes do not elute within the defined range during calibration verification, the instrument must be recalibrated and new windows defined. New studies are performed when major changes, such as column replacement are made to the chromatographic system.

11.0 INSTRUMENT MAINTENANCE

***Requirement*.** Procedures must be established for equipment maintenance. The procedure may include a maintenance schedule if required or documentation of daily maintenance related activities. All instrument maintenance activities must be documented in instrument specific logbooks. All equipment out of service (both analytical and auxiliary) must be clearly marked "Out of Order".

11.1 ***Routine, Daily Maintenance***. Routine, daily maintenance is required on an instrument specific basis. It is performed each time the instrument is used. Daily maintenance traditionally includes activities to insure a continuation of good analytical performance. In some cases, they include performance checks that indicate whether non-routine maintenance is required. If the performance check indicates a need for higher level maintenance, the equipment is taken out of service until maintenance is performed. Analysis cannot be continued until the performance checks meet established criteria. Document return to control. Daily maintenance is the responsibility of the individual assigned to the instrument used for the analysis he is performing.

11.2 ***Non-routine Maintenance***. Non-routine maintenance is reserved for catastrophic occurrences such as instrument failure. The need for non-routine maintenance is indicated by failures in general operating systems that result in an inability to conduct required performance checks or calibration. Equipment in this category are taken out of service and repaired before attempting further analysis. Analysis cannot continue until the instrument meets all performance check criteria and is capable of being calibrated. Section supervisors are responsible for identifying non-routine maintenance episodes and initiating repair activities to bring the equipment on-line. This may include initiating telephone calls to maintenance contractors if necessary. They are also responsible for documenting all details related to the occurrence and the repair.

11.3 ***Scheduled Maintenance***. Modern laboratory instrumentation rarely requires regular preventative maintenance. Where required, the equipment is placed on a schedule, which dictates when maintenance is required. Examples include annual balance calibration by an independent provider and optical alignment of the ICP. Section supervisors are responsible for initiating scheduled maintenance on equipment that requires scheduled preventative attention. Scheduled maintenance is documented using routine documentation practices.

11.4 ***Maintenance Documentation***. Routine and non-routine maintenance activities are documented in logbooks assigned to instruments and equipment used for analytical measurements. The logbooks contain preprinted forms, which specify the maintenance activities required with each use. Accutest Laboratories Southeast has adopted a problem – action – follow-up format to conduct instrument maintenance. The analyst or supervisor who performs or initiates the maintenance activity is required to check the activity upon its completion, verify complete statement of return to normal conditions and initial the form. Non-routine maintenance (i.e. repairs, upgrades, etc.) is documented as well either electronically via e-mail from the service provider or receipt attached to the maintenance log.

12.0 QUALITY CONTROL PARAMETERS, PROCEDURES, AND CORRECTIVE ACTION

Requirement: All procedures used for test methods must incorporate quality control parameters to monitor elements that are critical to method performance. Each quality parameter includes acceptance criteria that have been established by regulatory agencies for the methods in use. Criteria may also be established through client dictates or through the accumulation and statistical evaluation of internal performance data. Data obtained from these parameters must be evaluated by the analyst, and compared to established method criteria. If the criteria are not achieved, the procedures must specify corrective action and conformation of control before proceeding with sample analysis. QC parameters, procedures, and corrective action must be documented within the standard operating procedures for each method. In the absence of client specific objectives the laboratory must define qualitative objectives for completeness and representativeness of data.

12.1 ***Procedure***. Bench analysts are responsible for methodological quality control and sample specific quality control. Each method specifies the control parameters to be employed for the method in use and the specific procedures for incorporating them into the analysis. These control parameters are analyzed and evaluated with every designated sample group (batch).

The data from each parameter provides the analyst with critical decision making information on method performance. The information is used to determine if corrective action is needed to bring the method or the analysis of a specific sample into compliance. These evaluations are conducted throughout the course of the analysis. Each parameter being indicative of a critical control feature. Failure of a methodological control parameter is indicative of either instrument or batch failure. Failure of a sample control parameter is indicative of control difficulties with a specific sample or samples.

Sample Batch. All samples analyzed in the laboratory are assigned to a designated sample batch, which contains all required quality control samples and a defined maximum number of field samples that are prepared and/or analyzed over a defined time period. The maximum number of investigative and field QC samples in the batch is 20. Accutest has incorporated the NELAP batching policy as the sample-batching standard. This policy incorporates the requirement for blanks and spiked blanks as a time based function as defined by NELAP. The typical batch contains a blank, laboratory control sample (LCS or spiked blank), matrix spike and matrix spike duplicate. Batch documentation includes lot specifications for all reagents and standards used during preparation of the batch.

12.2 ***Methodological Control Parameters and Corrective Action***. Prior to the analysis of field sample the analyst must determine that the method is functioning properly. Specific control parameters indicate whether critical processes meet specified requirements before continuing with the analysis. Method specific control parameters must meet criteria before sample analysis can be conducted. Each of these

parameters is related to processes that are under the control of the laboratory and can be adjusted if out of control.

Method Blank. A method blank is analyzed during the analysis of any field sample. The method blank is defined as a sample. It contains the same standards (internal standards, surrogates, matrix modifiers, etc.) and reagents that are added to the field sample during analysis, with the exception of the sample itself. If the method blank contains target analyte(s) at concentrations that exceed method or client requirements (typically defined as 1/2 RL concentrations), the source of contamination is eliminated before proceeding with sample analysis. Systematic contamination is documented for corrective action and resolved following the established corrective action procedures. In specific cases, contamination detected in the method blank may be acceptable if the concentrations do not exceed regulatory limits or client defined reporting limits.

Laboratory Control Samples (LCS or Spiked Blanks). A laboratory control sample (spiked blank or commercially prepared performance evaluation sample) is analyzed along with field samples to demonstrate that the method accuracy is within acceptable limits. These spike solutions are derived from different sources than the solutions used for method calibration. The performance limits are derived from published method specifications or from statistical controls generated from laboratory method performance data. Spiked blanks are blank matrices (reagent water or clean sand) spiked with the targeted parameters and analyzed using the same method used for samples. Accuracy data is compared to laboratory experimentally derived limits to determine if the method is in control. Laboratory control samples (LCS) are commercially prepared spiked samples in an inert material. Performance criteria for recovery of spiked analytes is pre-established by the commercial entity preparing the sample. This sample is analyzed in the laboratory as an external reference.

Accuracy data is compared to the applicable performance limits. If the spike accuracy exceeds the performance limits, corrective action, as specified in the SOP for the method is performed and verified before continuing with a field sample analysis. In some cases, decisions are made to continue with sample analysis if performance limits are exceeded; provided the unacceptable result has no negative impact on the sample data.

Marginal exceedance (ME) values are calculated for methods containing more than eleven (11) targeted analytes. The ME is calculated as $\pm$ 4 standard deviations about the mean. MEs are considered for multi-analyte methods because of the increased likelihood of LCS failure as the number of analytes in the method increase. The number of allowable MEs is based on the number of target analytes in the method. Analytes that regularly fall into the ME category are treated as systematic problems, which are resolved using established trend monitoring and corrective action procedures. Marginal Exceedances are not applied to parameters that are detected in field samples. Routine corrective action is initiated for all cases where LCS spike accuracy criteria is beyond the established control limits and the parameter is detected in field samples corresponding to the unacceptable LCS.

Blanks and spikes are routinely evaluated before samples are analyzed. However, in situations where sample analysis is performed using an autosampler, they may be evaluated after sample analysis has occurred. If the blanks and spikes do not meet criteria, sample analysis is repeated.

Proficiency Testing. Performance Evaluation (Proficiency Testing) samples (PEs, PTs) are single or double blind samples spiked with know amount of analytes on interest and introduced to the laboratory to assess method performance. PEs may be introduced as double blinds submitted by commercial clients, single or double blinds from regulatory agencies, or internal blinds submitted by the QA group.

A minimum of two single blind studies must be performed each year for every parameter in aqueous and solid matrices for each field of proficiency testing (FOPT) for which the laboratory maintains accreditation. Proficiency Testing samples must be purchased as blinds from an accredited vendor. Data from these studies are provided to the laboratory by the vendor and reported to accrediting agencies. If unsatisfactory performance is noted, corrective action is performed to identify and eliminate any sources of error. A new PT must be analyzed to demonstrate continuing proficiency.

PE samples performed for accrediting agencies or clients, which do not meet performance specifications, require a written summary that documents the corrective action investigation, findings, and corrective action implementation.

Single or double blind PT samples are employed for self-evaluation purposes. Data from these analyses are compared to established performance limits. If the data does not meet performance specifications, the system is evaluated for sources of acute or systematic error. If required, corrective action is performed and verified before initiating or continuing sample analysis.

Trend Analysis for Control Parameters. Accuracy data for selected spiked parameters from the laboratory control sample (LCS) is statistically evaluated daily for trends. Data from selected LCS parameters and surrogates are pooled on a method, matrix, and instrument basis. This data is evaluated by comparison to existing control and warning limits. Trend analysis is performed automatically as follows:

- Any point outside the control limit
- Any three consecutive points between the warning and control limits
- Any eight consecutive points on the same side of the mean
- Any six consecutive points increasing or decreasing

The results of the trend analysis are printed for supervisory evaluation prior to sample analysis. Trends that indicate the potential loss of statistical control are further evaluated to determine the impact on data quality and to determine if corrective action is necessary. If corrective action is indicated, the supervisor informs the analysts of

the corrective actions to be performed. Return to control is demonstrated before analysis resumes.

12.3 ***Sample Control Parameters and Corrective Action***. The analysis of samples can be initiated following a successful demonstration that the method is operating within established controls. Additional controls are incorporated into the analysis of each sample to determine if the method is functioning within established specifications for each individual sample. Sample QC data is evaluated and compared to established performance criteria. If the criteria are not achieved the method or the SOP specifies the corrective action required to continue sample analysis. In many cases, failure to meet QC criteria is a function of sample matrix and cannot be remedied. Each parameter is designed to provide quality feedback on a defined aspect of the sampling and analysis episode.

Duplicates. Duplicate sample analysis is used to measure analytical precision. This can also be equated to laboratory precision for homogenous samples. Precision criteria are method dependent. If precision criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Laboratory Control Duplicate, Spikes & Spiked Duplicates. Spikes and spiked duplicates are used to measure analytical precision and accuracy for the sample matrix selected. Precision and accuracy criteria are method dependent. If precision and accuracy criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Serial Dilution (Metals). Serial dilutions of metals samples are analyzed to determine if analytical matrix effects may have impacted the reported data. If the value of the serially diluted samples does not agree with the undiluted value within a method-specified range, the sample matrix may be causing interference, which may lead to either a high or low bias. If the serial dilution criterion is not achieved, it must be flagged to indicate possible bias from matrix effects. *Accutest-SE uses this procedure as opposed to post-digestion spike unless contractual obligations absolutely require latter*

Post Digestion Spikes. Digested samples are spiked and analyzed to determine if matrix interferences are creating biases in the results. It may also be used to determine potential interferences per client's specification. Spike concentration is determined as per analytical method. No action is necessary if the post digestion spike is outside of the method criteria, unless a preparation problem is suspected with the spike, in which case the post digestion spike should remade and reanalyzed.

Surrogate Spikes (Organics). Surrogate spikes are organic compounds that are similar in behavior to the target analytes but unlikely to be found in nature. They are added to all quality control and field samples to measure method performance for each individual sample. Surrogate accuracy limits are derived from published method

specifications or by statistical evaluation of laboratory generated surrogate accuracy data. Accuracy data is compared to the applicable performance limits. If the surrogate accuracy exceeds performance limits, corrective action, as specified in the method or SOP is performed before sample data can be reported.

Internal Standards (Organic Methods). Internal standards are retention time and instrument response markers added to every sample to be used as references for quantitation. Their response is compared to reference standards and used to evaluate instrument sensitivity on a sample specific basis. Internal standard retention time is also compared to reference standards to assure that target analytes are capable of being located by their individual relative retention time.

If internal standard response criteria are not achieved, corrective action or additional action may be required. The recommended action must be completed before sample data can be reported.

If the internal standard retention time criteria are not achieved corrective action or additional action may be required. This may include re-calibration and re-analysis. Additional action must be completed before sample data is reported.

Internal Standards (ICP Metals). Internal standards are used on ICP instruments to compensate for variations in response caused by differences in sample matrices. This adjustment is performed automatically during sample analysis. The internal standard response of replicated sample analysis is monitored to detect potential analytical problems. If analytical problems are suspected, then the field samples are reanalyzed.

12.4 ***Laboratory Derived Quality Control Criteria.*** Control criteria for in-house methods and client specific modifications that exceed the scope of published methodology are defined and documented prior to the use of the method. The Quality Assurance staff identifies the responsibility for control criteria needs. Control parameters and criteria, based on best technical judgement are established using input provided by the operations staff. These control parameters and criteria are documented and incorporated into the method.

The laboratory derived criteria are evaluated for technical soundness on spiked samples prior to the use of the method on field samples. The technical evaluation is documented and archived by the Quality Assurance staff.

When sufficient data form the laboratory developed control parameter is accumulated, the data is statistically processed and the experimentally derived control limits are incorporated into the method.

12.5 ***Bench Review & Corrective Action***. The bench chemists are responsible for all QC parameters. Before proceeding with sample analysis, they are required to successfully meet all instrumental QC criteria. They have the authority to perform any necessary corrective action before proceeding with sample analysis. Their authority

includes the responsibility for assuring that departures from documented policies and procedures do not occur.

The bench chemists are also responsible for all sample QC parameters. If the sample QC criteria are not achieved, they are authorized and required to perform the method specified corrective action before reporting sample data.

***Data Qualifiers*.** An alpha character coding system is employed for defining use limitations for reported data. These limitations are applied to analytical data by the analyst to clarify the usefulness of the reported data for data user. Accutest Laboratories Southeast qualifies data in accordance with program-specific requirements, such as State of Florida DEP, AFCEE, etc., and these qualifiers are hard-coded in the LIMS on project level. Definitions of common qualifiers could be found at the bottom of the sample report form.

12.6 ***QA Monitoring***. The QA staff prior to client release conducts a spot review of completed data packages. This review includes an examination of QC data for compliance and trends indicative of systematic difficulties. If non-conformances are detected, the QA staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation. The data package is released when the package becomes compliant with all quality requirements.

If the review reveals trends indicative of systematic problems, QA initiates an investigation to determine the cause. If process defects are detected, a corrective action is implemented and monitored for effectiveness.

Performance Limits. The Technical Director is responsible for compilation and maintenance of all precision and accuracy data used for performance limits. Quality control data for all test methods are accumulated and stored in the laboratory information management system (LIMS). Parameter specific QC data is extracted annually and statically processed to eliminate outliers and develop laboratory specific warning limits and confidence limits. The new limits are reviewed and approved by the supervisory staff prior to their use for data assessment. The new limits are used to evaluate QC data for compliance with method requirements for a period of one year. Laboratory generated limits appear on all data reports unless method specifies hard-coded limits (mostly General Chemistry and Metals)

12.7 ***Data Package Review***. Accutest employs multiple levels of data review to assure that reported data has satisfied all quality control criteria and that client specifications and requirements have been met. Production departments have developed data review procedures which must be conducted before data is released to the client.

Analytical Review. The analyst conducts the primary review of all data. This review begins with a check of all instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. Analyst checks focuses on a review of qualitative

determinations and checks of precision and accuracy data to verify that existing laboratory criteria have been achieved. Checks at this level may include comparisons with project specific criteria if applicable. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of review.

Secondary data reviews are performed at the peer level by analysts who have met the qualification criteria for the method in use. Qualification requirements include a valid demonstration of capability and demonstrated understanding of the method SOP. Section supervisors may perform secondary review in-lieu of a peer review Secondary review is performed on 100% of the data produced by their department. It includes a check of all manual calculations; an accuracy check of manually transcribed data from bench sheets to the LIMS, a check of all method and instrument QC criteria, baseline manipulations (if applicable) and a comparison of the data package to client specified requirements. Also included are checks to assure the appropriate methodology was applied and that all anomalous information was properly flagged for communication in the case narrative. Supervisors have the authority to reject data and initiate re-analysis, corrective action, or reprocessing.

All laboratory data requiring manual entry into LIMS system is double-checked by the analysts performing initial data entry and the section supervisor. Verification of supervisory review is indicated on the raw data summary by the supervisor's initials and date.

Electronic data that is manually edited at the bench by the primary analysts is automatically flagged by the instrument data system indicating an override by the analyst. All manual overrides must be verified and approved by a supervisor who initials and dates all manual changes.

Hard copies of manually integrated chromatographic peaks are printed that clearly depict the manually drawn baseline. The hard copy is reviewed and approved by the reviewer (initialed and dated) and included in the data package of all full tier reports or the archived batch records of commercial report packages.

Electronic data that has been committed to the LIMS can only be edited by a manager or supervisor. These edits may be required if needs for corrections are indicated during the final review. An audit record for all electronic changes in the LIMS is automatically appended to the record.

The group manager performs a tertiary review on a spot check basis. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

Report Generation Review. The report generation group reviews all data and supporting information delivered by the laboratory for completeness and compliance

with client specifications. Missing deliverables are identified and obtained from the laboratory. The group also reviews the completed package to verify that the delivered product complies with all client specifications. Non-analytical defects are corrected before the package is sent to the client.

Project Management/Quality Assurance Review. Spot-check data package reviews are performed by the project manager. Project management reviews focus on project specifications. If the project manager identifies defects in the product prior to release, he initiates immediate corrective action to rectify the situation.

The QA Staff reviews approximately 10% of the data produced. The QA review focuses on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification. QA reviews at this step in the production process are geared towards systematic process defects, which require procedural changes to effect a corrective action. However, if defects are identified that can be corrected prior to data release, the QA staff returns the package to the laboratory for corrective action. QA data review cannot be used in lieu of a peer level review or a supervisory review.

Data Reporting. Analytical data is released to clients following secondary departmental review. Data release at this stage of the process is limited to electronic information, which is released to clients through a secure, encrypted, password protected, Internet connection.

Hard copy support data is compiled by the report generation group and assembled into the final report. The report is sent to the client following reviews by report generation, and spot-check by QA staff.

All data reports include specified information, which is required to identify the report and its contents. This information includes a title, name and address of the laboratory, a unique report number, total number of pages in the report, clients name and address, analytical method identification, arriving sample condition, sample and analysis dates, test results with units of measurement, authorized signature of data release, statement of applicability, report reproduction restrictions and TNI requirements certification. Subcontracted data is clearly identified.

In the event of report revision date of the revision, nature of revision and identity of the person revising the report must be clearly stated in the body of the report. Depending on the level of the deliverables it could be either stated in the Case Narrative or Case Narrative generated specifically for this purpose. Case Narrative must state "supercedes all previous reports".

12.8 ***Electronic Data Reduction.*** Raw data from sample analysis is entered into the laboratory information management system (LIMS) using automated processes or manual entry. Final data processing is performed by the LIMS using procedures developed by the Company.

All LIMS programs and internally developed software (including Excel spreadsheets) are tested and validated prior to use to assure that they consistently produce correct results. Validation testing is performed by the Information Technology Staff. The testing procedures are documented in an SOP. Programs are not approved for use until they have demonstrated that they are capable of performing the required calculations.

12.9 ***Representativeness***. Data representativeness is based on the premise that qualitative and quantitative information developed for field samples is characteristic of the sample that was collected by the client and analyzed in the laboratory. The laboratory objective for representativeness defines data as representative if the criteria for all quality parameters associated with the analysis of the sample are achieved.

12.10 ***Comparability***. Analytical data is defined as comparable when data from a sample set analyzed by the laboratory is representatively equivalent to other sample sets analyzed separately regardless of the analytical logistics. The laboratory will achieve 100% comparability for all sample data which meets the criteria for the quality parameters associated with its analysis using the method requested by the client.

13.0 CORRECTIVE ACTION SYSTEM

Requirement. The laboratory must have polices and procedures for correcting defective processes, systematic errors, and quality defects, which enables the staff to systematically improve product quality. The system must include procedures for communicating items requiring corrective action, corrective action tracking procedures, corrective action documentation, monitoring of effectiveness, and reports to management. The system must be documented in a standard operating procedure.

13.1 ***Procedure***. Corrective action is the step that follows the identification of a process defect. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance.

Routine Corrective Action. Routine corrective action is defined as the procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters or analytical system specifications.

Bench analysts have full responsibility and authority for performing routine corrective action. The resolution of defects at this level does not require a procedural change or staff re-training. The analyst is free to continue work once corrective action is complete and the analytical system has been returned to control. Documentation of routine corrective action is limited to bench logbook or maintenance logbook comment.

Process Changes. Corrective actions in this category require procedural modifications. They may be the result of systematic defects identified during audits, the investigation of client inquiries, failed proficiency tests, product defects identified during data review, or method updates. Resolution of defects of this magnitude requires formal identification of the defect, development and documentation of a corrective action plan, and staff training to communicate the procedural change.

Technical Corrective Action. Technical corrective action encompasses routine corrective action performed by bench analysts for out of control systems and corrective actions performed for data produced using out of control systems. Technical corrective action for routine situations is conducted using the procedures detailed above.

Non-routine corrective actions apply to situations where the bench analysts failed to perform routine corrective action before continuing analysis. Supervisors and Department Managers perform corrective action in these situations. Documentation of all non-routine corrective actions is performed using the corrective action system.

Sample re-analysis is conducted if sufficient sample and holding time remain to repeat the analysis using an in-control system. If insufficient sample or holding time remains, the data is processed and qualifiers applied that describe the out of control situation. The occurrence is further documented in the case narrative and in the corrective

action response. The corrective action must include provisions for retraining the analysts who failed to perform routine corrective action.

13.2 ***Documentation & Communication***. Routine corrective actions are documented as part of the analytical record. Notations are made in the comments section of the analytical chronicle or data sheet detailing the nonconformance. Continuation of the analysis indicates that return to control was successful.

Corrective actions for process changes are documented, tracked and monitored for effectiveness. Corrective actions may be initiated by any supervisor or senior staff member by completing the corrective action form in Corrective Action database

The corrective action database is an Access application. The initiator generates the corrective action investigation form, which is documented, tracked, distributed to responsible parties and archived through the application. The application assigns a tracking number initiation data and due date to each corrective action initiated and copies the corrective action form to the corrective action database. The application also distributes an E-mail message containing the form to the responsible parties for resolution.

Corrective Action system employs Deficiency – Root Cause – Immediate Fix – Corrective action approach, further divided into categories of Analytical Error, Omission Error, Random Error, Systemic Error and Training Issue.

The responsible party develops and implements the procedural change. Existing documentation such as SOPs are edited to reflect the change. The affected staff is informed of the procedural change through a formal training session. The training is documented and copies are placed into individual training files. The corrective action form is completed and closed in CA database.

Initial and completed corrective action forms are maintained in the Corrective Action directory. This information is archived daily. Copies of training records describing corrective actions are appended to the involved individuals training files.

Monitoring. The QA Staff monitors the implemented corrective action until it is evident that the corrective action has been effective and the systematic deficiency has been eliminated. The corrective action database is updated by QA to reflect closure of the corrective action. The QA staff also assigns an error code to the corrective action for classification of the type of errors being committed.

If QA determines that the corrective action procedure has not effectively remedied the deficiency, the process continues with a re-initiation of the corrective action. Corrective action continues until the defective process is eliminated. If another procedural change is required, it is treated as a new corrective action, which is documented and monitored using established procedures.

Client Notification. Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, Accutest will notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.

14.0 PROCEDURES FOR EXECUTING CLIENT SPECIFICATIONS

***Requirement*.** Systems must be established for evaluating and processing client specifications for routine and non-routine analytical services. The systems must enable the client services staff to identify, evaluate, and document the requested specifications to determine if adequate resources are available to perform the analysis. The system must include procedures for communicating the specifications to the laboratory staff for execution and procedures for verifying the specifications have been executed.

14.1 ***Client Specific Requirements***. The project manager is the primary contact for clients requesting laboratory services. Client specifications are communicated using several mechanisms. The primary source of information is the client's quality assurance project plan (QAPP) which details analytical and quality control specifications for the project. In the absence of a QAPP, projects specifications can also be communicated using contracts, letters of authorization, or letters of agreement, which may be limited to a brief discussion of the analytical requirements and the terms and conditions for the work. These documents may also include pricing information, liabilities, scope of work, in addition to the analytical requirements. QAPPs include detailed analytical requirements and data quality objectives, which supersede those found in the referenced methods. This information is essential to successful project completion.

Laboratory also reviews its Accreditation status to evaluate whether it is possible to accept proposed project. Discrepancies must be resolved before the work commences.

The client services staff provides additional assistance to clients who are unsure of the specifications they need to execute the sampling and analysis requirements of their project. They provide additional support to clients who require assistance in results interpretation as needed, provided they possess the expertise required to render an opinion.

The project manager is responsibility for obtaining project documents, which specify the analytical requirements. Following project management review, copies are distributed to the QA staff and the appropriate departmental managers for review and comment. The original QAPP is numbered with a document control number and filed in a secure location.

14.2 ***Requirements for Non-Standard Analytical Specifications***. Client requirements that specify departures from documented policies, procedures, or standard specifications must be submitted to Accutest in writing. These requirements are reviewed and approved by the technical staff before the project is accepted. Once accepted, the non-standard requirements become analytical specifications, which follow the routine procedure for communicating client specifications. Departures from documented policies, procedures, or standard specifications that do not follow this procedure are not permitted.

Exception Policy: With respect to the quality system, incoming non-conforming product refers to received samples that do not meet requirements of custody documentation, are improperly packaged or stored or are contaminated. An internal non-conformance refers to a problem, caused internally due to improper handling of samples, improper sampling methods, and equipment malfunction or data management errors. The individual who identifies the incoming non-conformance is responsible for notifying the project manager. The project manager resolves the issue with the client. The individual who recognizes an internal non-conformance is responsible for initiating corrective action

Departures from standard practices, policies and specifications are reviewed and approved by Technical Director, QA Officer and by Project Manager of the project affected.

Corrective & Preventative Action: Once a quality problem has been identified, the analytical or review process stops, until the reason is identified. Primary responsibility for identifying the cause of the problem rests with the instrument operator. Other staff may be called on to assist in reaching the root cause. The problem prevention tracking system, using Corrective Action Tracking Records, provides a method to track systemic problems until resolved/removed. The QA Officer is responsible for the record management with respect to the disposition of problems.

Deviations that do not limit themselves to a single department and/or client are cited on Corrective Action Record. This may include but not limited to: sample arrival outside of EPA specified holding time, analysis completion outside of EPA specified holding time (with explanation of the reason), inconsistencies between chain of custody and cooler contents, including labeling errors, improper preservation, etc.

Deviations from analytical methods' SOP's are reported by the Analyst to the Section Leader. Single occurrences warrant completion of Corrective Action Tracking record, repetitive occurrences may indicate that either an additional training session is in order, or the SOP does not reflect proper laboratory practice. Training session is conducted by the Technical Director or by QA Officer. In case where SOP does not reflect current laboratory practice, SOP review and correction process may be initiated.

14.3 ***Evaluation of Resources.*** A resource evaluation is completed prior to accepting projects submitted by clients. The evaluation is initiated by the client services staff receives project requirements (usually in the form of QAPjP) and distributes these requirements to the laboratory departments affected. The specifications are evaluated by the department managers from a scheduling and hardware resources perspective. The project is not accepted unless the department managers have the necessary resources to execute the project according to client specifications.

14.4 ***Documentation***. New projects are initiated using a project set up form, which is completed prior to the start of the project. This form details all of the information needed to correctly enter the specifications for each client sample into the laboratory information management system (LIMS, see example). The form includes data reporting requirements, billing information, data turnaround times, QA level, state of origin, and comments for detailing project specific requirements. The project manager is responsible for obtaining this information from the client and completing the form prior to sample arrival and login.

Sample receipt triggers project creation and the login process. The information on the set-up form is entered into the LIMS immediately prior to logging in the first sample. The set up form may be accompanied by a quotation, which details the analytical product codes and sample matrices. These details are also entered into the LIMS during login.

Special information is distributed to the laboratory supervisors and login department in electronic or hardcopy format upon project setup. All project specific information is retained by the project manager in a secure file. The project manager maintains a personal telephone log, which details conversations with the client regarding the project.

14.5 ***Communication***. A pre-project meeting is held between client services and the operations managers to discuss the specifications described in the QAPjP and/or related documents. Project logistics are discussed and finalized and procedures are developed to assure proper execution of the client's analytical specifications and requirements. Questions, raised in the review meeting, are discussed with the client for resolution. Exceptions to any requirements, if accepted by the client, are documented and incorporated into the QAPjP or project documentation records.

Non-standard specifications for individual clients are documented in the LIMS at the client account level. Once entered into the LIMS, these specifications become memorialized for all projects related to the client account. Upon sample arrival, these specifications are accessed through a terminal or printed as a hard copy and stored in a binder for individuals who require access to the specification. Specifications that are not entered into the LIMS are prohibited unless documented in an interdepartmental memo, which clearly identifies the project, client and effective duration of the specification.

14.6 ***Operational Execution***. A work schedule is prepared for each analytical department on a daily basis. Analytical specifications from recently arrived samples have now been entered into the LIMS database. The database is sorted by analytical due date and holding time, into product specific groups. Samples are scheduled for analysis by due date and holding time. The completed schedule, which is now defined as a work list, is printed. The list contains the client requested product codes and specifications required for the selected sample(s). Special requirements are communicated to the analyst using the comments section or relayed through verbal instructions provided by

the supervisor. The bench analyst assumes full responsibility for performing the analysis according to the specifications printed on the work sheet.

14.7 ***Verification***. Prior to the release of data to the client, laboratory section managers and the report generation staff review the report and compare the completed product to the client specifications documentation to assure that all requirements have been met. Project managers perform a spot check of projects with unique requirements to assure that the work was executed according to specifications.

15.0 CLIENT COMPLAINT RESOLUTION PROCEDURE

Requirement. A system for managing and reconciling client complaints must be implemented in the laboratory. The system must include procedures for documenting client complaints and communicating the complaint to the appropriate department for resolution. The system must also include a quality assurance evaluation to determine if the complaint is related to systematic defects requiring process changes.

15.1 ***Procedure***. Client complaints are communicated to client services representatives, quality assurance staff, or senior management staff for resolution. The individual receiving the complaint retains the responsibility for documentation and communicating the nature of the complaint to the responsible department(s) for resolution. The responsible party addresses the complaint. The resolution is communicated to quality assurance (QA) and the originator for communication to the client. QA reviews the complaint and resolution to determine if systematic defects exist. If systematic defects are present, QA works with the responsible party to develop a corrective action that eliminates the defect.

Documentation. Client's complaints are documented by the client service representative receiving the complaint. A record of the telephone conversation is maintained by client services. Client service staff enters the complaint into Data Challenge database or Client Complaint database, depending on the nature of complaint. These databases are cross-linked with corrective action database – see sec. 13. Complaint is communicated to the production departments concerned via auto e-mail. The complaint resolution is documented in the database by the responsible party and resultant e-mail returned to the originator. QA staff is copied on the correspondence.

15.2 ***Corrective Action***. Responses to Data Challenges/Client Complaints are required from the responsible party. At a minimum, the response addresses the query and provides an explanation to the complaint. Corrective action may focus on the single issue expressed in the complaint. Corrective action may include job case narrative generation, reprocessing of data, editing of the initial report, and re-issue to the client. If the QA review indicates a systematic error, process modification is required. The defective process at the root of the complaint is changed. SOPs are either created or modified to reflect the change. The party responsible for the process implements process changes.

15.3 ***QA Monitoring***. Process changes, implemented to resolve systematic defects, are monitored for effectiveness by QA. If monitoring indicates that the process change has not resolved the defect, QA works with the department management to develop and implement an effective process. If monitoring indicates that the defect has been resolved, monitoring is slowly discontinued. Continued monitoring is incorporated as an element of the annual system audit and annual Management Report (see 18.8).

16.0 CONTROL OF NONCONFORMING PRODUCT

Requirement: Policies and procedures have been developed and implemented that describe the procedures employed by the laboratory when any aspect of sample analysis or data reporting do not conform to established procedures or client specifications. These procedures include steps to ensure that process defects are corrected and affected work is evaluated to assess its impact to the client.

Procedure. Nonconforming product is identified through multiple channels, such as second level analytical data review, routine internal review and audit practices, external auditing or through client inquiry. Responsibility and authority for the management of the non-conforming product directly defined by a nature of a non-conformance. For example, non-conformances resulting from internal and external reviews are evaluated and managed by QA Staff. Corrective Action items are issued and followed to completion and verification that defect is prevented from reoccurring. Non-conformances stemming from client inquiry are managed by Project Management staff with QA staff oversight.
Data associated with out-of compliance QC are evaluated by bench personnel and section supervisors. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of review.
If non-conformances are detected, the QA staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation

Non-conformances and their significance are communicated in case narrative and sample report footnotes. Case narrative comments and sample repot footnotes must state the impact on data quality.

Corrective Action. The outcome of the evaluation dictates the course of action. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance This may include at a minimum client notification, but may also include corrective action. Immediate corrective action is performed using the SOP-specified procedures. However, additional action may be required including cessation of analysis and withholding and/or recalling data reports. If the evaluation indicates that nonconforming data may have been issued to clients, the client is immediately notified and data may be recalled following the procedures specified in respective SOPs. If work has been stopped because of a nonconformance, the Laboratory Director is the only individual authorized to direct a resumption of analysis.
Nonconformances caused by systematic process defects require retraining of the personnel involved as an element of the corrective action solution. Routine corrective actions are documented as part of the analytical record.

17.0 CONFIDENTIALITY PROTECTION PROCEDURES

Requirements: Policies and procedures are required to protect client data from release to unauthorized parties or accidental release of database information through accidental electronic transmission or illegal intrusion. These policies must be communicated to clients and staff. Electronic systems must be regularly evaluated for effectiveness.

17.1 ***Client Anonymity***. Information related to the Company's clients is granted to employees on a "need to know" basis. An individual's position within the organization defines his "need to know". Individuals with "need to know" status are given password access to systems that contain client identity information and access to documents and document storage areas containing client reports and information. Access to client information by individuals outside of the Company is limited to the client and individuals authorized by the client.

Individuals outside of the Company may obtain client information through subpoena issued by a court of valid jurisdiction. Clients are informed when subpoenas are received ordering the release of their information.

17.2 ***Documents***. Access to client documents is restricted to employees in need to know positions. Copies of all client reports are stored in secure archive with restricted access. Reports and report copies are distributed to individuals who have been authorized by the client to receive them. Documents are not released to third parties without verbally expressed or written permission from the client.

17.3 **Confidential Business Information (CBI).** Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between Accutest and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as Adobe Acrobat .PDF format only. See also Sec. 6.5.

17.4 ***Electronic Data***.

Database Intrusion. Direct database entry is authorized for employees of Accutest only on a need to know basis. Entry to the database is restricted through a user specific multiple password entry system. Direct access to the database outside of the facility is possible through a VPN connection. A unique password is required for access to the local area network. A second unique password is required to gain access to the database. The staff receives read or write level authorization on a hierarchical privilege basis.

Internet Access. Access to client information is through an HTTP Web application only. It does not contain a mechanism that allows direct access to the database. Clients can gain access to their data only using a series of Accutest assigned accounts, and client specific passwords. The viewable data, which is encrypted during transmission, consists of an extraction of database information only.

Client Accessibility. Accessibility to client data delivered via electronic means follows strict protocols to insure confidentiality. Clients accessing electronic data are assigned a company account. The account profile, which is established by the MIS staff, grants explicit access to explicit information pertaining to the clients project activity. Passwords are assigned on an individual basis within a client account. These accounts can be activated or deactivated by the MIS staff only.

17.5 ***Information Requests***. Client specific data or information is not released to third parties without verbally expressed or written permission from the client. Written permission is required from third parties, who contact the Company directly for the release of information. Verbal requests will be honored only if they are received directly from the client. These requests must be documented in a record of communication maintained by authorized recipient.

17.6 ***Transfer of Records***. Archived data, which has previously been reported and transmitted to clients, is the exclusive property of Accutest Laboratories. In the event of a cessation of business activities due to business failure or sale, The Company's legal staff will be directed to arrange for the final disposition of archived data.

The final disposition of archived data will be accomplished using the approach detailed in the following sequence:

1. All data will be transferred to the new owners for the duration of the required archive period as a condition of sale.

2. If the new owners will not accept the data or the business has failed, letters will be sent to clients listed on the most recent active account roster offering them the option to obtain specific reports (identified by Accutest Job Number) at their own expense.

3. A letter will be sent to the TNI accrediting authority with organizational jurisdiction over the company offering them the option to obtain all unclaimed reports at their own expense.

4. All remaining archived data will be recycled using the most expedient means possible.

18.0 QUALITY AUDITS AND SYSTEM REVIEWS

Requirement: The quality assurance group will conduct regularly scheduled audits of the laboratory to assess compliance with quality system requirements, technical requirements of applied methodology, and adherence to documentation procedures. The information gathered during these audits will be used to provide feedback to senior management and perform corrective action where needed for quality improvement purposes.

18.1 ***Quality Systems Review***. Quality system audits are performed annually by the Quality Assurance Director for the Company President. In this audit, the laboratory is evaluated for compliance with the Laboratory Quality Systems Manual (LQSM) and the quality system standards of the National Environmental Laboratory Accreditation Conference. Findings, which indicate non-compliance or deviation from the LQSM, are flagged for corrective action. Corrective actions require either a return to compliance or a plan change to reflect an improved quality process. The QA Officer is responsible for making and documenting changes to the LQSM. These changes are reviewed by the Laboratory Director and Technical Director prior to the approval of the revised system.

18.2 ***Quality System Audits.*** Quality system audits are conducted to evaluate the effectiveness and laboratory compliance with individual quality system elements. These audits are conducted on an established schedule. Audit findings are documented and communicated to the management staff and entered into the corrective action system for resolution. If necessary, retraining is conducted to assure complete understanding of the system requirements.

18.3 ***Technical Compliance Audits***. Technical compliance audits are performed throughout the year following the established schedule. Selected analytical procedures are evaluated for compliance with standard operating procedures (SOPs) and method requirements. If non-conformances exist, the published method serves as the standard for compliance. SOPs are edited for compliance if the document does not reflect method requirements. Analysts are trained to the new requirements and the process is monitored by quality assurance. Analysts are retrained in method procedures if an evaluation of bench practices indicates non-compliance with SOP requirements.

18.4 ***Documentation Audits***. Documentation audits are conducted periodically. This audit includes a check of measurement processes that require manual documentation and non-analytical logbook review. It also includes checks of data archiving systems and a search to find and remove any inactive versions of SOPs that may still be present in the laboratory and being accessed by the analysts. Non-conformances are corrected on the spot. Procedural modifications are implemented if the evaluation indicates a systematic defect.

18.5 ***Corrective Action Monitoring***. Defects or non-conformances that are identified during client or internal audits are shared with management and entered into CA

database for attention by the responsible party. Audit findings are corrected through process modifications and/or retraining. Once a corrective action has been designed and implemented, it is monitored for compliance on a regular basis by the QA staff. Monitoring of the corrective action continues until satisfactory implementation has been verified.

18.6 ***Preventive Action.*** Laboratory systems or processes, which may be faulty and pose the potential for nonconformances, errors, confusing reports or difficulties establishing traceability may be identified during internal audits. These items are highlighted for systematic change using the corrective action system and managed to resolution using appropriate procedures for corrective action.

18.7 ***Client Notification.*** Defective processes, systematic errors, and quality defects detected during routine audits may have negative impact on data quality. In some cases, data that has been released to the client may be affected. If defective data has been released for use, Accutest will immediately notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.

18.8 ***Management Reports***. Formal reports of all audit activities are prepared for the management staff. These reports are prepared annually. The report details the status of the Quality System

The formal report also addresses the following topics:

- *the suitability of policies and procedures;*
- *reports from managerial and supervisory personnel;*
- *the outcome of recent internal audits;*
- *corrective and preventive actions;*
- *assessments by external bodies;*
- *the results of interlaboratory comparisons or proficiency tests;*
- *changes in the volume and type of the work;*
- *customer feedback;*
- *complaints;*
- *recommendations for improvement;*
- *other relevant factors, such as quality control activities, resources, and staff training.*

19.0 HEALTH AND SAFETY

Requirement. The company operates a formal health and safety program that complies with the requirements of the Occupational Health and Safety Administration. The program consists of key policies and practices that are essential to safe laboratory operation. All employees are required to receive training on the program elements. Job specific training is conducted to assure safe practices for specific tasks. All employees are required to participate in the program, receive initial and annual training, and comply with the program requirements. All plan and program requirements are detailed in the Health and Safety Program Manual.

19.1 **Policy.** Accutest Laboratories will provide a safe and healthy working environment for its employees and clients while protecting the public and preserving the Company's assets and property. The company will comply with all applicable government regulations pertaining to safety and health in the laboratory and the workplace.

The objective of the Accutest Health and Safety Program is to promote safe work practices that minimize the occurrence of injuries and illness to the staff through proper health and safety training, correct laboratory technique application and the use of engineering controls.

19.2 **Responsibilities.** The Health and Safety Program assists managers, supervisors and non-supervisory employees in control of hazards and risks to minimize the potential for employee and client injuries, damage to client's property and damage or destruction to Accutest's facility.

The Health and Safety Officer is responsible for implementing the Program's elements and updating its contents as necessary. He also conducts periodic audits to monitor compliance and assess the program's effectiveness and is also responsible for creating and administering safety training for all new and existing employees.

The employee is responsible for following all safety rules established for their protection, the protection of others and the proper use of protective devices provided by the Company. The employee is also expected to comply with the requirements of the program at all times. Department Managers and Supervisors are responsible for ensuring the requirements of the Safety Program are practiced daily. The Company President retains the ultimate responsibility for the program design and implementation.

19.3 **Program Elements.** The Accutest Health and Safety Program consists of key program elements that compliment the company's health and safety objective. These elements form the essence of the health and safety policy and assure that the objectives of the program are achieved.

Safety Education and Training and Communication. Training is conducted to increase the staff's awareness of laboratory hazards and their knowledge of the safety

practices and procedures required to protect them from those hazards. It is also used to communicate general safety procedures required for safe operation in a chemical laboratory.

Initial health and safety training for new employees is conducted during orientation. The training focuses on the Accutest Safety and Health Program and includes specific training for the hazards that may be associated with the employees' duties. Training is also conducted for all program elements focusing on general, acceptable, laboratory safety procedures. Targeted training is conducted to address hazards or safety procedures that are specific to individual employee's work assignments. All training activities are documented and archived in individual training folders. A health and safety training inventory is maintained in the training database.

Accutest Laboratories Southeast maintains personnel trained in HAZWOPER, DOT and HazMat operations, as well as respirator certified.

Safety Officer. The safety officer provides the employees with an opportunity to express their views and concerns on safety issues in an environment where those concerns will be addressed to ensure that the interests of the company and the well being of the employee are protected. Safety Officer is entrusted with elevating the level of safety awareness among their peers.

***Hazard Identification and Communication*.** The hazard communication program enables employees to readily identify laboratory hazards and the procedures to protect themselves from those hazards. This program complies with OSHA's Hazard Communication Standard, Title 29 Code of Federal Regulations 1910.1200 that requires the company to adopt and adhere to the following key elements:

- Material Safety Data Sheets (MSDS) and/or Safety Data Sheets (SDS) must be available to any employee wishing to view them,
- The Company must maintain a Hazardous Chemicals Inventory (by location), which is updated on an annual basis,
- Containers are properly labeled,
- All employees must be provided with annual Personal Protection, Hazard Communication and Right to Know training,

Chemical Hygiene Plan. The Chemical Hygiene Plan complies with the requirements of the Occupational Safety and Health Administration's Occupational Exposure to Hazardous Chemicals in the Laboratory Standard, 29 CFR 1910.1450. This plan establishes procedures, identifies safety equipment, personal protective equipment, and work practices that protect employees from the potential health hazards presented by hazardous chemicals in the laboratory if properly used and/or applied.

Emergency Action & Evacuation Plan. The Emergency Action and Evacuation Plan details the procedures used to protect and safeguard Accutest's employees and property during emergencies. Emergencies are defined as fires or explosions, gas leaks, building collapse, hazardous material spills, emergencies that immediately threaten life and health, bomb threats and natural disasters such as floods, hurricanes or tornadoes. The plan identifies and assigns responsibility for executing specific roles in situations requiring emergency action.

Lockout/Tagout Plan. Lockout/tagout procedures have been established to assure that laboratory employees and outside contractors take steps to render equipment inoperable and/or safe before conducting maintenance activities. The plan details the procedures for conducting maintenance on equipment that has the potential to unexpectedly energize, start up, or release energy or can be operated unexpectedly or accidentally resulting in serious injury to employees. The plan ensures that employees performing maintenance render the equipment safe through lock out or tag out procedures.

Personal Protection Policy. Policies have been implemented which detail the personal protection requirements for employees. The policy includes specifications regarding engineering controls, personal protective equipment (PPE), hazardous waste, chemical exposures, working with chemicals and safe work practices. Safety requirements specific to processes or equipment are reviewed with the department supervisor or the Health and Safety Officer before beginning operations.

Emergency Preparedness Plan. This plan identifies the actions to be taken by Accutest Laboratory's staff in the event of terrorism or terrorist actions, to ensure the safety of the employees and the facility. The plan describes the building security actions coinciding with the "Alert Condition", designated by the Department of Homeland Security.

Appendix I

Glossary of Terms

GLOSSARY OF TERMS

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents.

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.

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.

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.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

Audit: a systematic evaluation to determine the conformance to quantitative *and qualitative* specifications of some operational function or activity.

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 to 20 environmental samples of the same quality-system 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 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 environmental matrices and can exceed 20 samples.

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 sub-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.

Case Narrative: a statement of non-conformances associated with particular data report

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.

Calibration Curve: the mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Method: a defined technical procedure for performing a calibration.

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.

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.

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 to enforce them.

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.

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.

Conformance: an affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

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).

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.

Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy.

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 is performed.

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples 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.

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.

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 submit to only that portion of the accreditation process not previously addressed (see TNI, section 1.9ff).

Holding Times (Maximum Allowable Holding Times) the maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

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 from a source independent of the calibration standards or a material containing known and verified amounts of analytes. 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.

Matrix (or Quality System 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 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, Biota: 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 sorbent tube, impinger solution, filter, or other device.

Matrix Spike (spiked sample or fortified sample): a 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 Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which 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.

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.

National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, TNI, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater.

The NELAC institute (TNI): a voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories.

TNI 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 The NELAC Institute.

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.

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.

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.

PT Fields of Testing: TNI's approach to offering proficiency testing by regulatory or environmental program, matrix type, and analyte.

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.

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.

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.

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality Manual: a document stating the management policies, objectives, principles, oganizational 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.

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.

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.

Range: the difference between the minimum and the maximum of a set of values.

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof 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 media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank or method 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.

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.

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

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.

Requirement: denotes a mandatory specification; often designated by the term “shall”.

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.

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.

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.

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.

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 TNI procedures and policies.

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.

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.

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.

Validation: the process of substantiating specified performance criteria.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.
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.

Appendix II

Analytical Capabilities

TNI-Accredited Fields of Testing

Method Type	Method Number	Regulatory Program
Organics		
EDB and DBCP	EPA 504.1	Drinking Water
1,4-Dioxane	EPA 522	Drinking Water
Metals		
ICP: General	EPA 200.7, 1994	Drinking Water
Cold Vapor Mercury	EPA 245.1, 1994	Drinking Water
Inorganic WetChem		
Perchlorate by Ion Chromatography	EPA 314.0	Drinking Water
Organics		
EDB and DBCP	EPA 504, SW846 8011**	Non-Potable Water
Volatile Organics	EPA 624, SW846 8260B**	Non-Potable Water
Semi-Volatile Organics	EPA 625, SW846 8270D**	Non-Potable Water
Semi-Volatile Organics	SW846 8270D SIM**	Non-Potable Water
Purgeable Aromatics	EPA 602, SW846 8021A**	Non-Potable Water
Chlorinated Pesticides & PCBs	EPA 608, SW846 8081B**, 8082A**	Non-Potable Water
Poly-Aromatic Hydrocarbons	EPA 610, SW846 8310**	Non-Potable Water
Nitroaromatics	SW846 8091**	Non-Potable Water
Explosives	SW846 8330A**, 8332**	Non-Potable Water
Explosives	SW846 8330B**,	Non-Potable Water
Chlorinated Herbicides	SW846 8151A**	Non-Potable Water
Organophosphorus Pesticides	SW846 8141B**	Non-Potable Water
Perchlorate	SW-846 6850	Non-Potable Water
Dissolved Gases	RSK SOP 147-175**	Non-Potable Water
Alcohols	SW846 8015C,D**	Non-Potable Water
Gasoline Range Organics	SW846 8015C,D**	Non-Potable Water
Diesel Range Organics	SW846 8015C,D**	Non-Potable Water
Total Petroleum Hydrocarbons	FLPRO**	Non-Potable Water
Tennessee EPH	TN-EPH**	Non-Potable Water
Tennessee GRO	TN-GRO**	Non-Potable Water
Wisconsin DRO	WI-DRO**	Non-Potable Water
Petroleum Hydrocarbons	Iowa OA-1**	Non-Potable Water
Petroleum Hydrocarbons	Iowa OA-2**	Non-Potable Water
Volatile Petro. Hydrocarbons	Massachusetts VPH, 2004**	Non-Potable Water

Method Type	Method Number	Regulatory Program
Extractable Petro. Hydrocarbons	Massachusetts EPH, 1998**	Non-Potable Water
Total Petroleum Hydrocarbons	TX-1005**	Non-Potable Water
Acrylamide	SW846 8316	Non-Potable Water
Metals		
ICP: General – EPA WW	EPA 200.7, 1994; SW-846 6010C**	Non-Potable Water
Cold Vapor Mercury – EPA WW	EPA 245.1, 1994; SW-846 7470A**	Non-Potable Water
Inorganic WetChem		
Alkalinity	SM2320B**	Non-Potable Water
CBOD	SM 5210B	Non-Potable Water
COD	SM5220C	Non-Potable Water
BOD	SM5210B	Non-Potable Water
Color, Apparent	SM2120B	Non-Potable Water
Ion Chromatography (Bromide, Fluoride, Chloride, Sulfate, Nitrite, Nitrate,) – Aqueous	EPA 300.0**, SW846 9056A**	Non-Potable Water
Nitrate/Nitrite	EPA 353.2**	Non-Potable Water
Total Kjeldahl Nitrogen	EPA 351.2**	Non-Potable Water
Ammonia	EPA 350.1**	Non-Potable Water
Oil & Grease, Gravimetric – AQ	EPA 1664A**, SW846 9070A**	Non-Potable Water
Orthophosphate	EPA 365.3**	Non-Potable Water
Nitrate	SM 4500NO2-B	Non-Potable Water
pH by electrode (Waters)	SM4500H+B**; SW846 9040C**	Non-Potable Water
Specific Conductance	EPA 120.1	Non-Potable Water
Nitrate-Nitrite	SM 4500 NO3-E	Non-Potable Water
Sulfide	SM4500S=F**	Non-Potable Water
Chloride	SM 4500 Cl-B	Non-Potable Water
Total Dissolved Solids	SM2540C**	Non-Potable Water
Total Organic Carbon	SM5310B**, SW846 9060A**	Non-Potable Water
Total Phosphorus	EPA 365.3	Non-Potable Water
Total Solids	SM2540B**	Non-Potable Water
Total Suspended Solids	SM2540D**	Non-Potable Water
Turbidity	EPA 180.1	Non-Potable Water
Total CN	EPA 335.4, SW846 9012B**	Non-Potable Water
Un-Ionized Ammonia - calculation	FDE SOP10/03/83	Non-Potable Water
Perchlorate	EPA 314	Non-Potable Water
Calcium Hardness by Calculation	SM18 2340B	Non-Potable Water
Hardness, Total by Calculation	SM18 2340B	Non-Potable Water
MBAS (Anionic Surfactants as)	SM5540C	Non-Potable Water

Method Type	Method Number	Regulatory Program
Corrosivity & pH – aqueous	SW846 9040C**	Non-Potable Water
Hexavalent Chromium	SW846 7196A**	Non-Potable Water
Organics		
EDB and DBCP	SW846 8011 Mod**	Solid and Chemical Material
Volatile Organics	SW846 8260B**	Solid and Chemical Material
Semi-Volatile Organics	SW846 8270D**	Solid and Chemical Material
Semi-Volatile Organics	SW846 8270D SIM**	Solid and Chemical Material
Gasoline Range Organics	SW846 8015C,D**	Solid and Chemical Material
Diesel Range Organics	SW846 8015C,D**	Solid and Chemical Material
Alcohols	SW846 8015C,D**	Solid and Chemical Material
Polynuclear-Aromatic Hydrocarbons	SW846 8310**	Solid and Chemical Material
Explosives	SW846 8330A**, 8332**	Solid and Chemical Material
Explosives	SW846 8330B**	Solid and Chemical Material
Organochlorine Pesticides	SW846 8081B**	Solid and Chemical Material
Polychlorinated Biphenyls	SW846 8082A**	Solid and Chemical Material
Chlorinated Herbicides	SW846 8151A**	Solid and Chemical Material
Organophosphorus Pesticides	SW846 8141B**	Solid and Chemical Material
Perchlorate	SW-846 6850	Solid and Chemical Material
Total Petroleum Hydrocarbons	FLPRO**	Solid and Chemical Material
Tennessee EPH	TN-EPH**	Solid and Chemical Material
Tennessee GRO	TN-GRO**	Solid and Chemical Material
Wisconsin DRO	WI-DRO**	Solid and Chemical Material
Petroleum Hydrocarbons	Iowa OA-1**	Solid and Chemical

Method Type	Method Number	Regulatory Program
		Material
Petroleum Hydrocarbons	Iowa OA-2**	Solid and Chemical Material
Volatile Petro. Hydrocarbons	Massachusetts VPH, 2004**	Solid and Chemical Material
Extractable Petro. Hydrocarbons	Massachusetts EPH, 1998**	Solid and Chemical Material
Total Petroleum Hydrocarbons	TX-1005**	Solid and Chemical Material
Acrylamide	SW846 8316	Solid and Chemical Material
Metals		
ICP: General – EPA WW	SW846 6010C**	Solid and Chemical Material
Cold Vapor Mercury – EPA DW	SW846 7471B**	Solid and Chemical Material
Inorganic WetChem		
Ion Chromatography (Bromide, Fluoride, Chloride, Sulfate, Nitrite, Nitrate,) – Aqueous	SW846 9056A**	Solid and Chemical Material
Oil & Grease, Gravimetric – Solid	SW846 9071A**	Solid and Chemical Material
Total CN	SW846 9012B**	Solid and Chemical Material
Total Organic Carbon	SW846 9060A**	Solid and Chemical Material
Ammonia	EPA 350.1	Solid and Chemical Material
Total Kjeldahl Nitrogen	EPA 351.2	Solid and Chemical Material
Total Phosphorus	EPA 365.3	Solid and Chemical Material
Waste Ignitability	SW846 1010A**	Solid and Chemical Material
Hexavalent Chromium/soils	SW846 7196A**	Solid and Chemical Material
Corrosivity & pH – aqueous	SW846 9040C**	Solid and Chemical Material
Corrosivity & pH – solid	SW846 9045D**	Solid and Chemical Material

Method Type	Method Number	Regulatory Program
Cyanide Reactivity	SW846 Chapter 7**	Solid and Chemical Material
Sulfide Reactivity	SW846 Chapter 7**	Solid and Chemical Material
Organics		
Volatile Organics	TO-3	Air and Emissions
Preparation Methods*		
Liquid/Liquid Extraction, Water	SW846 3510C	
Solid Phase Extraction, Water	SW846 3535A	
Solids Extraction by Sonication	SW846 3550B	
Microwave-assisted extraction, solids	SW846 3546	
Acid/Base Partitioning	SW846 3650B	
Sulfur Cleanup of Extracts	SW846 3660B	
Sulfuric Acid Cleanup	SW846 3665A	
Purge & Trap - Aqueous	SW846 5030B	
Purge & Trap – Solids	SW846 5035A	
Total Recoverable Metals Digestion	EPA 200.7	
Non-Pot. Water Digest: ICP	SW846 3010A	
Alkaline Digestion of Soils for Hexavalent Chromium	SW846 3060A	
Digestion of Soils for ICP	SW846 3050B	
TCLP	SW846 1311	
SPLP	SW846 1312	

* Preparation methods are not listed on Primary TNI Accreditation per State of Florida DOH rules. However, for the benefit of other accrediting authorities, these methods are inspected during FDOH visits. Listing of surveyed and approved preparation methods is available from on-site inspection report.
** Methods certified by DoD ELAP

Non-TNI-Accredited Fields of Testing

Method Type	Method Number	Regulatory Program
Organics		
Thiodiglycol	Accutest in-house method (HPLC)	Solid and Chemical Material
N-Nitroso-N-Ethylurea	Accutest in-house method (HPLC)	Solid and Chemical Material
Volatile Petroleum Hydrocarbons	Missouri Gasoline Range Organics	Solid and Chemical Material
Extractable Hydrocarbons	Missouri Diesel Range Organics	Solid and Chemical Material
Extractable Hydrocarbons	Missouri Oil Range Organic	Solid and Chemical Material
Volatile Petroleum Hydrocarbons	Alaska AK-101**	Solid and Chemical Material
Extractable Hydrocarbons	Alaska AK-102**	Solid and Chemical Material
Extractable Hydrocarbons	Alaska AK-103**	Solid and Chemical Material
Volatile Petroleum Hydrocarbons	OK GRO**	Solid and Chemical Material
Extractable Hydrocarbons	OK DRO**	Solid and Chemical Material
Inorganic WetChem		
Oxidation-Reduction Potential	ASTM D1498-76, mod. for solids	Solid and Chemical Material
Percent Ash (dry basis)	ASTM D2974-87, D482-91	Solid and Chemical Material
Grain Size (hydrometer)	ASTM D422-63	Solid and Chemical Material
Sieve Testing	ASTM D422-63	Solid and Chemical Material
Specific Gravity	ASTM D1298-85	Solid and Chemical Material
Acidity	SM2310B	Non-Potable Water
Dissolved Oxygen	EPA 360.1	Non-Potable Water
Mineral Suspended Solids	EPA 160.2/160.4	Non-Potable Water
Organophosphonic Acids	Accutest in-house method (IC)	Solid and Chemical Material

Method Type	Method Number	Regulatory Program
Perchlorate	EPA 314MOD	Solid and Chemical Material
Percent Solids	SM19 2540G	Solid and Chemical Material
Settleable Solids	EPA 160.5	Non-Potable Water
Total Mineral Solids	EPA 160.4	Non-Potable Water
Total Residual Chlorine	EPA 330.5	Non-Potable Water
Total Volatile Solids	EPA 160.4	Non-Potable Water
Volatile Suspended Solids	EPA 160.2/160.4	Non-Potable Water
CÑ Amenable to Chlorination	EPA 335.4	Solid and Chemical Material
Bicarbonate, Carbonate, CO2 - calculation	SM19 4500 CO2D	Non-Potable Water
Ferrous Iron	SM19 3500 FE-D	Non-Potable Water
Salinity - calculation	SM19 2520B	Non-Potable Water
Paint Filter Test	SW846 9095	Solid and Chemical Material
Corrosivity towards steel	SW846 1110	Solid and Chemical Material
Corrosivity & pH – aqueous	SW846 9040C	Solid and Chemical Material

Appendix III

Equipment List

ORGANIC INSTRUMENTATION

Instrument	Model	Location	Serial #	Year
GC/MS	Agilent 5975C MSD/OI 4551/4660	MS-VOA	US11172705	2011
GC/MS	Agilent 5975C MSD/OI 4551/4660	MS-VOA	US11322911	2011
GC/MS	Agilent 5975C MSD/OI 4551/4660	MS-VOA	US10102029	2010
GC/MS	Agilent 5975C MSD/OI 4551/4660	MS-VOA	US83120965	2008
GC/MS	Agilent 5975N MSD/Agilent 7683 AS	SVOC Lab	US71225975	2007
GC/MS	Agilent 5975N MSD/Agilent 7683 AS	SVOC Lab	US62724401	2006
GC/MS	Agilent 5975N MSD/Agilent 7683 AS	SVOC Lab	US53921303	2005
GC/MS	Agilent 5973N MSD/Agilent 7683 AS	SVOC Lab	US40620599	2004
GC/MS	Agilent 5973 MSD/OI 4660/4552 Archon	MS-VOA	US41746628	2004
GC/MS	Agilent 5973 MSD/OI 4660/4552 Archon	MS-VOA	US41746633	2004
GC/MS	Agilent 5973 MSD/OI 4560/4552 Archon	Soil VOA	US21843765	2002
GC/MS	Agilent 5973 MSD/OI 4551/4660	MS-VOA	US21844034	2002
GC/MS	Agilent 5973 MSD/OI 4660/4552 Archon	MS-VOA	US02440350	2000
GC/MS	Agilent 5973 MSD/OI 4560/4552 Archon	MS-VOA	US94240108	1999
GC/MS	Agilent 5973 MSD/Agilent 7683 AS	SVOC Lab	US82311290	1998
GC/MS	Agilent 5973 MSD/Agilent 7683 AS	SVOC Lab	US81211109	1998
GC/MS	Hewlett-Packard 5970 MSD/OI 4560/4552 Archon	Soil VOA	3034A12782	1989
GC/MS	Hewlett-Packard 5970 MSD/OI 4560/4552 Archon	Soil VOA	2905A11904	1987
GC/MS	Hewlett-Packard 5970 MSD/OI 4560/4552 Archon	Soil VOA	2716A10454	1987
GC	Agilent 7890A/Dual ECD/7683B AS	SVOC Lab	CN10842133	2008
GC	Agilent 7890A/Dual FID/7683B AS	SVOC Lab	CN10902149	2009
GC	Agilent 7890A/Dual FID/7683B AS	SVOC Lab	CN10716029	2009
GC	Agilent 7890A/Dual ECD/7683B AS	SVOC Lab	CN10741128	2007

Instrument	Model	Location	Serial #	Year
GC	Agilent 6890/Dual FPD/7683B AS	SVOC Lab	US10643024	2006
GC	Agilent 6890/Dual FID/7683B AS	SVOC Lab	CN10641049	2006
GC	Agilent 6890/Dual ECD/7683B AS	SVOC Lab	CN10641081	2006
GC	Agilent 6890/Dual ECD/7683B AS	SVOC Lab	US10613003	2006
GC	Agilent 6890/PID/PID/OI 4560/4552 Archon	GC VOA	CN10421047	2004
GC	Agilent 6890/PID/FID/ENTECH 7032A-LB	GC VOA	US10239007	2002
GC	Agilent 6890N/Dual FID/HP 7683 AS	SVOC Lab	CN10425061	2004
GC	Agilent 6890N/Dual ECD/HP 7683 AS	SVOC Lab	US10333015	2003
GC	Agilent 6890/Dual ECD/HP 7683 AS	SVOC Lab	US00036916	2000
GC	Agilent 6890/Dual ECD/HP 7683 AS	SVOC Lab	US00028304	1999
GC	Hewlett-Packard 5890/PID/FID/ OI 4560/4552 Archon	GC VOA	3336A60617	1993
GC	Hewlett-Packard 5890/Dual FID/HP 7673 AS	SVOC Lab	3336A59489	1993
GC	Hewlett-Packard 5890/PID/FID/ OI 4560/4552 Archon	GC VOA	3336A51045	1993
GC	Hewlett-Packard 5890/PID/FID/OI 4560/4552 Archon	GC VOA	3203A41646	1992
GC	Hewlett-Packard 5890/PID/FID/OI 4560/4552 Archon (screening instrument)	GC VOA	3223A4267	1992
GC	Hewlett-Packard 5890/Dual FID/HP 7673 AS	SVOC Lab	3126A51085	1991
GC	Hewlett-Packard 5890/PID/FID/ dual MPM 16	Soil VOA	3029A29748	1990
GC	Hewlett-Packard 5890/FID	Soil VOA	2843A20183	1988
GC	Hewlett-Packard 5890/Dual FID	GC VOA	2728A12705	1987
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE91606857	1999
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE23917648	2002
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE01608404	2000
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE40522115	2004
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE03000863	2003

Instrument	Model	Location	Serial #	Year
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE61800775	2006
O-Prep	ESSA LM2-P Ring and Puck mill	Explosives Prep Lab	215090-004	2008
O-prep	Microwave extractor	Organic Prep Lab	MD3482	2010
O-Prep	TurboVap 4 units	Organic Prep Lab		2001
O-Prep	TurboVap 3 units	Organic Prep Lab		2004
O-Prep	TurboVap 1 unit	Organic Prep Lab		2007
O-Prep	Sonicator 2 units	Organic Prep Lab		2004
O-Prep	Sonicator 3 units	Organic Prep Lab		2007
O-Prep	Midi-Vap 2000 Kontes	Organic Prep Lab	479200-2000	2000
Data System	Hewlett-Packard/MS ChemStation	Labwide		1999, with subsequent upgrades

Inorganic Instrumentation

Instrument	Model	Location	Serial #	Year
ICP	Thermo ICAP 6000 Series	Metals Lab	20100903	2010
ICP	Thermo ICAP 6000 Series	Metals Lab	20103825	2010
Mercury Analyzer	Leeman Hydra AA	Metals Lab	HA-2022	2002
Mercury Analyzer	Leeman Hydra AA II	Metals Lab	2004	2012
TOC Analyzer	Shimadzu	WetChem IC room	H51404235007	2004
TOC Analyzer	Shimadzu	WetChem IC room	H51404735099	2010
IC	Dionex IC-2100	WetChem IC room	10110002	2010
IC	Dionex IC-2000	WetChem IC room	04070250	2004
Auto Analyzer	QuickChem 8500 Series	WetChem main room	050500000130	2005
Auto Analyzer	QuickChem 8500 Series 2	WetChem main room	111200001380	2011
Spectrophotometer	Milton-Roy Spectronic 200	WetChem main room	2 units	2000
Digestion block	DigiPrep	WetChem main room	4 units	2005

Centrifuge	CentraCL2	WetChem main room	42613052	2003
MicroDistillation Block	Lachat	WetChem main room	2 units	2005

LIMS		
Instrument	**Model**	**Year**
LIMS	HP True 64	1999

Appendix IV

Certification Summary

Certifying Authority	**Certification Program**	**Registration No.**
Alaska	Contaminated Sites	UST-088
Arkansas	Solid/Hazardous Wastes, Non-Potable Water	88-0620
California (NELAP)	Potable Water, Solid/Hazardous Waste	04226CA
Department of Defense (DoD)	Non-Potable Water, Solid and Chemical Materials	L-2229
Florida (NELAP)	Potable, Non-Potable, Solid Waste, UST, Air Toxics	E83510
Georgia	Solid/Hazardous Wastes	Not Applicable
Illinois	Solid/Hazardous Wastes, Non-Potable Water	
Iowa	UST, Solid/Hazardous Wastes, Non-Potable Water	IA366
Kansas (NELAP)	Solid/Hazardous Wastes, Non-Potable Water	E-10327
Kentucky	Underground Storage Tank Program	0065
Louisiana (NELAP)	Solid/Hazardous Wastes	38582
Massachusetts	Non-Potable Water	M-FL946
Mississippi	Potable Water	Not Applicable
Nevada	Non-Potable Water, Solid/Hazardous Wastes	FL009462008A
New Jersey (NELAP)	Solid/Hazardous Wastes, Non-Potable Water	FL002
North Carolina	Solid/Hazardous Wastes, Non-Potable Water	573
Oklahoma	Non-Potable Water, Solid/Hazardous Waste	9959
South Carolina	Solid/Hazardous Wastes, Non-Potable Water	96038001
Texas (NELAP)	Non-Potable Water, Solid/Hazardous Waste	T104704040-08-TX
US Dept. of Agriculture	Foreign Soils Permit	S-56027
Utah (NELAP)	Potable, Non-Potable, Solid/Chemical Materials	FL009462008A
Virginia (NELAP)	Potable, Non-Potable, Solid/Chemical Materials	460177
Washington	Potable, Non-Potable, Solid/Chemical Materials, Air	C2046
Wisconsin	Solid/Hazardous Wastes, Non-Potable Water	399043370

Appendix V

SOP List

SOP #	TITLE
	Organic Preparation Department
OP002	SOP for Glassware Cleaning and Storage
OP003	SOP for Reagent Prep
OP006	SOP for the Extraction of Semi-volatile Organics (BNAs) from Aqueous Samples
OP007	SOP for the Extraction of Semi-volatile Organics (BNAs) from Solid Samples
OP008	SOP for the Extraction of Pesticides/PCBs from Aqueous Samples
OP009	SOP for the Extraction of Pesticides/PCBs from Solid Samples
OP009MW	SOP for the Extraction of Pesticides/PCBs from Solid Samples, microwave
OP010	SOP for the Extraction of Diesel Range Organics (DRO) from Aqueous Samples
OP011	SOP for the Extraction of Diesel Range Organics (DRO) from Solid Samples
OP011MW	SOP for the Extraction of Diesel Range Organics (DRO) from Solid Samples
OP012	SOP for the Extraction of Petroleum Related Organics (FL-PRO) from Aqueous Samples
OP013	SOP for the Extraction of Petroleum Related Organics (FL-PRO) from Solid Samples
OP014	SOP for the Extraction of PAHs from Aqueous Samples (HPLC)
OP015	SOP for the Extraction of PAHs from Solid Samples (HPLC)
OP016	SOP for the Extraction of EDB/DBCP from Aqueous Samples
OP017	SOP for the Extraction of EDB/DBCP from Solid Samples
OP018	SOP for the Extraction of Explosives from Aqueous Samples
OP019	SOP for the Extraction of Explosives from Solid Samples
OP020	SOP for Sample Introduction via SW846-5035
OP021	SOP for Sample Introduction via SW846-5030B
OP022	SOP For The Extraction Of Nitroglycerine And Pentaerythritoltetranitrate (PETN) From Water Samples (HPLC Analysis)
OP023	SOP For The Extraction Of Nitroglycerine And Pentaerythritoltetranitrate (PETN) From Solid Samples (HPLC Analysis)
OP024	Standard Operating Procedure For The Extraction Of Nitroaromatics From Water Samples
OP025	SOP For Sample Preparation For Dissolved Gases In Aqueous Samples
OP026	SOP For The Extraction Of Extractable Petroleum Products (OA-2) From Water Samples
OP027	SOP For The Extraction Of Extractable Petroleum Products (OA-2) From Solid Samples
OP028	SOP For The Extraction Of Diesel And Oil Range Organics From Water Samples
OP029	SOP For The Extraction Of Diesel And Oil Range Organics From Solid Samples
OP030	SOP For The Extraction Of Extractable Petroleum Hydrocarbons From

SOP #	TITLE
	Water Samples (Tennessee EPH)
OP031	SOP For The Extraction Of Extractable Petroleum Hydrocarbons From Solid Samples (Tennessee EPH)
OP032	SOP For The Extraction Of Volatile Petroleum Hydrocarbons From Soil Samples, MA-VPH
OP033	SOP For The Extraction Of PCBs From Wipes
OP034	SOP For The Extraction Of Diesel Range Organics (DRO) From Aqueous Samples WI-DRO
OP035	SOP For The Extraction Of Massachusetts Extractable Petroleum Hydrocarbons From Water Samples
OP036	SOP For The Extraction Of Massachusetts Extractable Petroleum Hydrocarbons From Solid Samples
OP037	SOP For The Extraction Of Chlorinated Herbicides From Water Samples
OP038	SOP For The Extraction Of Chlorinated Herbicides From Soil Samples
OP038MW	SOP For The Extraction Of Chlorinated Herbicides From Soil Samples, microwave
OP039	SOP For The Solid Phase Extraction (SPE) Cartridge Cleanup Of Pesticide Extracts
OP040	SOP For SPLP Leaching Of SVOC And Metals
OP041	SOP For TCLP Leaching Of VOC
OP042	SOP For SPLP Leaching Of SVOC And Metals
OP043	SOP For SPLP Leaching Of VOC
OP044	SOP For The Extraction Of Organophosphorus Pesticides From Water Samples
OP044SP	SOP For The Extraction Of Organophosphorus Pesticides From Water Samples, Solid Phase Extraction
OP045	SOP For The Extraction Of Organophosphorus Pesticides From Soil Samples
OP045MW	SOP For The Extraction Of Organophosphorus Pesticides From Soil Samples, microwave
OP046	SOP for the Extraction of Explosives from Solid Samples, SW-8330B
OP047	SOP for the Extraction of Explosives from Aqueous Samples, SW-8330B
OP048	SOP for the Extraction of PCB Congeners from Aqueous Samples
OP049	SOP for the Extraction of PCB Congeners from Solid Samples
OP050	SOP For The Extraction Of Alaska Extractable Petroleum Hydrocarbons From Water Samples
OP051	SOP For The Extraction Of Alaska Extractable Petroleum Hydrocarbons From Solid Samples
OP052	SOP For The Extraction Of Oklahoma Extractable Petroleum Hydrocarbons From Water Samples
OP053	SOP For The Extraction Of Oklahoma Extractable Petroleum Hydrocarbons From Solid Samples
OP054	SOP For The Extraction Of 1,4-Dioxane From Water Samples
OP055	SOP For The Extraction Of Petroleum Hydrocarbons From Water Samples,

SOP #	TITLE
	TX-1005
OP056	SOP For The Extraction Of Petroleum Hydrocarbons From Solid Samples, TX-1005
OP057	SOP for Sample Introduction via AK-101

Gas Chromatography/ HPLC SOPs

GC002	Analysis Of 1,2-Dibromoethane (EDB) And 1,2-Dibromo-3-Chloropropane (DBCP) By Gas Chromatography, Electron Capture Detector
GC004	Aromatic Volatiles By Gas Chromatography Using PID Detectors EPA 602
GC005	Analysis Of Organochlorine Pesticides By Gas Chromatography, Electron Capture Detector EPA 608
GC006	Analysis Of Polychlorinated Biphenyls By Gas Chromatography, Electron Capture Detector EPA 608
GC007	Analysis Of Polynuclear Aromatic Hydrocarbons By Gas Chromatography, Flame Ionization Detector EPA 610
GC008	Analysis Of Petroleum Range Organics By Gas Chromatography Using Flame Ionization Detector
GC009	Analysis Of 1,2-Dibromoethane (EDB) And 1,2-Dibromo-3-Chloropropane (DBCP) By Gas Chromatography, Electron Capture Detector SW-846 8011
GC010	Analysis Of Gasoline Range Organics By Gas Chromatography Using Flame Ionization Detector
GC011	Analysis Of Diesel Range Organics By Gas Chromatography Using Flame Ionization Detector
GC014	Analysis Of Polychlorinated Biphenyls By Gas Chromatography, Electron Capture Detector SW-846 8082
GC015	Analysis Of Organochlorine Pesticides By Gas Chromatography, Electron Capture Detector SW-846 8081
GC016	Analysis Of Nitroaromatics And Nitramines By HPLC
GC017	Aromatic Volatiles By Gas Chromatography Using PID Detectors SW-8021
GC018	Analysis Of Polynuclear Aromatic Hydrocarbons By HPLC SW-846 8310
GC019	Analysis Of Dissolved Gases By Gas Chromatography, Flame Ionization Detector
GC020	Analysis Of Nitroglycerine And PETN By HPLC
GC021	Analysis Of Volatile Petroleum Hydrocarbons By Gas Chromatography
GC022	Analysis Of Extractable Petroleum Products By Gas Chromatography Using Flame Ionization Detector OA-2
GC023	Analysis Of Diesel And Oil Range Organics By Gas Chromatography Using Flame Ionization Detector
GC024	Analysis Of Petroleum Hydrocarbons By Gas Chromatography Using Flame Ionization Detector (Tennessee EPH)
GC025	Analysis Of Nitroaromatics By Gas Chromatography Using Electron Capture Detector
GC026	Method For Determination Of Volatile Petroleum Hydrocarbons By GC-

SOP #	TITLE
	PID/FID
GC027	Analysis Of Non-Halogenated Organics By Gas Chromatography Using Flame Ionization Detector
GC028	Analysis Of Gasoline Range Organics By Gas Chromatography Using Flame Ionization Detector TDEC GRO
GC029	Analysis Of Diesel Range Organics By Gas Chromatography Using Flame Ionization Detector Wi DRO
GC030	Analysis Of Extractable Petroleum Hydrocarbons By Gas Chromatography Using Flame Ionization Detector MA-EPH
GC031	Analysis Of Chlorinated Herbicides Using GC-ECD
GC032	Analysis Of Organophosphorus Pesticides Using GC-NPD Or FPD
GC033	Air Analysis By GC-PID/FID
GC034	Analysis Of Nitroaromatics, Nitramines And Nitrate Esters By HPLC Method 8330b
GC035	Screening Of Volatile Organics By GC-PID/FID
GC036	Analysis of PCB Congeners by ECD
GC037	Analysis of Diesel and Oil Range Organics by GC/FID, AK-102, AK-103
GC038	Analysis of Gasoline Range Organics by GC/FID, AK-101
GC039	Analysis of Diesel Range Organics by GC/FID, OK-GRO
GC040	Analysis of Gasoline Range Organics by GC/FID, OK-GRO
GC041	Analysis of N-Nitroso-N-Ethylurea by HPLC
GC042	Analysis of Thiodiglycol by HPLC
GC043	Analysis of Acrylamide by HPLC
GC044	Analysis of Petroleum Organics by TX-1005

Mass-Spectrometry SOPs

MS003	Analysis of Volatile Organics by EPA Method 624
MS004	Analysis of Semi-volatile Organics by EPA Method 625
MS005	Analysis of Volatile Organics by EPA Method 8260B
MS006	Analysis of Semi-volatile Organics by EPA Method 8270C
MS008	Analysis of Semi-volatile Organics by EPA Method 8270C SIM
MS009	Analysis of Volatile Organics by GC/MS
MS010	Analysis of Volatile Organics by GC/MS SIM
MS011	Analysis of Semi-volatile Organics by EPA Method 8270D
MS012	Analysis of 1,4-Dioxane by EPA 522
MS013	Analysis of Perchlorate by SW-846 6850

Quality Assurance SOPs

QA001	Preparation, Approval, Distribution & Archiving Of Standard Operating Procedures (SOPs)
QA002	Calibration Of Thermometers
QA003	Personnel Training And Analyst Proficiency

SOP #	TITLE
QA004	Temperature Monitoring
QA005	Calibration Of Analytical Balances
QA006	Eppendorf Pipette Calibration
QA007	Sample Batching Procedure
QA008	Creating New Accounts
QA009	Creating New Projects
QA010	Confidentiality Protection Procedures
QA011	Signature Authority
QA012	Employee Technical Ethics Responsibilities
QA013	Client Complaint Resolution Procedure
QA014	Procedures For The Purchase Of Laboratory Supplies
QA015	Procedures For The Preparation, Distribution, Use And Archiving Of Laboratory Logbooks
QA016	Corrective Action Procedure
QA017	Standards Traceability Documentation Procedure
QA018	Procedure For Login, Management, Handling, And Reporting Of Proficiency Test (Pt) Samples
QA019	Quality System Review
QA020	Procedure For Developing Method Performance Criteria And Experimental Method Detection Limits
QA021	Subcontracting Procedures
QA022	Internal Audit Procedure
QA023	Fume Hood Inspection
QA027	Review Of Inorganics Data
QA028	Review Of Organics Data
QA029	Manual Integration Of Chromatographic Peaks
QA030	Procedure For The Development And Use Of in-house Quality Control Criteria
QA031	Air Quality Monitoring Of Extraction Laboratory
QA032	Routine Maintenance For Major Analytical Instrumentation
QA033	Laboratory Safety
QA034	Sample Homogenizing
QA035	Solvent Testing And Approval
QA036	Data Package Generation
QA037	Deionized Water Quality Control Procedure
QA038	Data Integrity Training Procedure
QA039	Data Integrity Monitoring Procedure
QA040	Procedure For Conducting Data Integrity Investigations
QA041	Procedure For The Confidential Reporting Of Data Integrity Issues
QA042	Basic Calculations For General Chemistry Methods
QA043	Data Qualifier SOP
QA044	Calibration Of Micro-Distillation Tubes
QA045	Estimation of Uncertainty
QA046	Document Control

SOP #	TITLE
QA047	Management of Client Project
QA048	Data Entry for Log-In

General Chemistry SOPs

GNSOP: 101	Acidity (pH 8.2)
GNSOP: 102	Alkalinity, Total (pH 4.5)
GNSOP: 103	Ammonia – Distillation Procedure
GNSOP: 104	Nitrogen, Ammonia
GNSOP: 105	Bicarbonate, Carbonate, Free Carbon Dioxide
GNSOP: 106	Chemical Oxygen Demand
GNSOP: 107	Chloride by Titration
GNSOP: 109	Color, Apparent
GNSOP: 110	Chromium, Hexavalent (Water)
GNSOP: 113	Cyanide Distillation/Aqueous And Solid Samples
GNSOP: 115	Cyanide, Total
GNSOP: 116	Dissolved Oxygen
GNSOP: 121	Ignitability
GNSOP: 122	Anionic Surfactants As MBAS
GNSOP: 123	Nitrogen, Nitrite
GNSOP: 126	Ortho Phosphate
GNSOP: 127	Paint Filter Liquids Test
GNSOP: 128	Phenols Distillation, Soil And Water Samples
GNSOP: 130	Phenols, Total Recoverable
GNSOP: 133	Settleable Solids
GNSOP: 134	Total Suspended Solids (Non Filterable Residue)
GNSOP: 135	Total Dissolved Solids (Total Filterable Residue)
GNSOP: 136	Reactive Sulfide And Reactive Cyanide
GNSOP: 137	pH By Electrode - Water
GNSOP: 140	Sulfide
GNSOP: 144	Total Phosphorus
GNSOP: 145	Turbidity
GNSOP: 147	Winkler Titration For DO Standardization
GNSOP: 161	Percent Solids
GNSOP: 163	Specific Conductance At 25 C.
GNSOP: 166	pH By Electrode – Soil
GNSOP: 167	Biochemical Oxygen Demand (BOD)
GNSOP: 171	Hexachromium In Soils
GNSOP: 179	Corrosivity (Soil pH By Electrode)
GNSOP: 182	Total Kjeldahl Nitrogen
GNSOP: 189	Corrosivity Toward Steel
GNSOP: 190	Total Nitrogen, Organic Nitrogen
GNSOP: 191	Nitrogen, Nitrate
GNSOP: 192	Carbonaceous Biochemical Oxygen Demand (CBOD)

SOP #	TITLE
GNSOP: 193	Oxidation-Reduction Potential
GNSOP: 194	Ferrous Iron
GNSOP: 196	Glassware Cleaning
GNSOP: 197	Anions By Ion Chromatography
GNSOP: 211	Oil & Grease And PHC By 1664
GNSOP: 212	Fractional Organic Carbon
GNSOP: 213	Walkley-Black Total Organic Carbon
GNSOP: 214	Particle Size By Sieve
GNSOP: 215	TOC In Water
GNSOP: 216	Particle Size By Hydrometer
GNSOP: 218	Perchlorate
GNSOP: 219	Bulk Density
GNSOP: 222	Un-Ionized Ammonia Calculation
GNSOP: 224	Hardness By Calculation
GNSOP: 225	Cation Exchange Capacity Of Soils (Sodium Acetate)
GNSOP: 226	TOC In Soil
GNSOP: 227	Oil And Grease – Gravimetric Analysis (Soils)
GNSOP: 228	Anions By Ion Chromatography - IC 2000
GNSOP: 229	Determination Of Nitrocellulose In Water
GNSOP: 230	Determination Of Nitrocellulose In Soil
GNSOP: 231	% Ash
GNSOP: 232	Determination Of Nitrate and Nitrite by Lachat

Metals SOPs

MET 100	Metals By Inductively Coupled Plasma
MET 103	Digestion Of Water Samples For Flame And ICP Analysis
MET 104	Digestion Of Soils For ICP Analysis
MET 105	Cold Vapor Analysis Of Mercury For Soils
MET 106	Cold Vapor Analysis Of Mercury For Water Samples

Sample Management SOPs

SAM101	Sample Receipt And Storage
SAM102	Procedure For Sample Bottle Preparation And Shipment
SAM104	Sample Container Quality Control
SAM108	Sample And Laboratory Waste Disposition
SAM109	Foreign Soil receipt and Handling

APPENDIX C
Project Schedule

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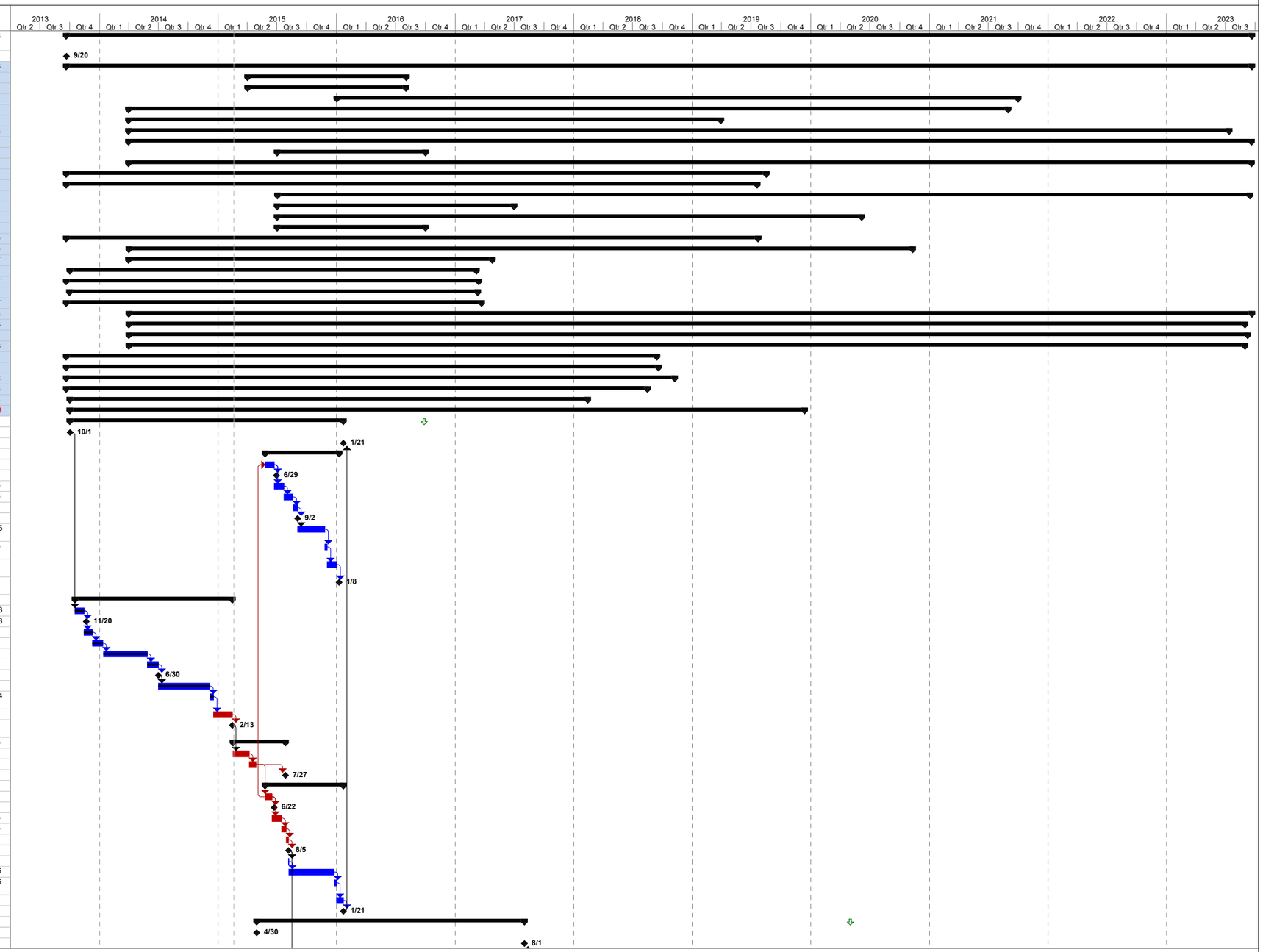
New Mexico-Arizona Group
Holloman AFB, New Mexico
Performance Based Remediation
Integrated Master Schedule

FPM Remediations, Inc.

ID	Site Name	SubCLIN	Name	Start	Finish
1			**Environmental Remediation in accordance with the PBR Statement of Objectives (SOO) - Contract Award**	**Fri 9/20/13**	**Wed 9/20/23**
2			Award	Fri 9/20/13	Fri 9/20/13
3			**Holloman AFB**	**Fri 9/20/13**	**Wed 9/20/23**
4	**Spill Area**		**SS013 - Sodium Arsenite Spill Area**	**Wed 4/1/15**	**Tue 8/2/16**
31	**Shop Area**		**OT014 - Former Entomology Shop Area**	**Wed 4/1/15**	**Mon 8/1/16**
58	**Fuel Spill**		**SS017 - BX Gas Station Fuel Spill**	**Fri 1/1/16**	**Thu 9/30/21**
250	**d Spill Site**		**SS018 - Chromic Acid Spill Site**	**Mon 3/31/14**	**Mon 8/30/21**
488	**Rad Waste**		**SD027 - PAD 9 Washrack Area - Rad Waste**	**Mon 3/31/14**	**Fri 3/29/19**
573	**my Landfill**		**LF029 - Old Army Landfill**	**Mon 3/31/14**	**Wed 7/12/23**
838	**posal Pits**		**DP030 - Grease Trap Disposal Pits**	**Mon 3/31/14**	**Tue 9/19/23**
1040	**earch Area**		**OT032 - Former Primate Research Area**	**Wed 7/1/15**	**Fri 9/30/16**
1067	**sal Trench**		**SD033 - Cooking Grease Disposal Trench**	**Mon 3/31/14**	**Tue 9/19/23**
1267	**esting Site**		**OT037 - Early Missile Testing Site**	**Fri 9/20/13**	**Fri 8/16/19**
1347	**ance Area**		**OT038 - Test Sled Maintenance Area**	**Fri 9/20/13**	**Fri 7/19/19**
1427	**Spill Area**		**SS039 - Missile Fuel Spill Area**	**Thu 7/2/15**	**Thu 9/14/23**
1759	**posal Area**		**RW042 - Radioactive Waste Disposal Area**	**Wed 7/1/15**	**Fri 6/30/17**
1864			**SS065 - Test Cell Surface Spill**	**Wed 7/1/15**	**Fri 6/5/20**
1924			**SS067 - Spill and Sand Blasting Residuals Area**	**Wed 7/1/15**	**Fri 9/30/16**
1951			**SS069 - Flightline Fuel Spill Area**	**Fri 9/20/13**	**Mon 7/22/19**
2012			**TU904 - Building 1194**	**Tue 4/1/14**	**Mon 11/9/20**
2170			**TU515 - Building 889**	**Mon 3/31/14**	**Mon 4/24/17**
2301			**TU503 - Building 221**	**Tue 10/1/13**	**Mon 3/6/17**
2432			**TU506 - Building 901**	**Fri 9/20/13**	**Mon 3/13/17**
2563			**TU508 - Building 298**	**Mon 9/30/13**	**Fri 3/10/17**
2688			**TU518 - UST 7003**	**Fri 9/20/13**	**Wed 3/22/17**
2819			**OD20K - 20,000 Pound Open Detonation Unit**	**Tue 4/1/14**	**Wed 9/20/23**
3032			**OW967 - SWMU 20 (Within T-38 Site)**	**Tue 4/1/14**	**Wed 8/30/23**
3233			**OW968 - SWMU 19 (Within T-38 Site)**	**Tue 4/1/14**	**Thu 9/7/23**
3434			**SS059, SS524 - T-38 Test Cell (SWMU 229)**	**Tue 4/1/14**	**Wed 8/30/23**
3635			**TS851 - Former Skeet Range**	**Fri 9/20/13**	**Thu 9/13/18**
3747			**XU853 - Missile Test Stands Area**	**Fri 9/20/13**	**Tue 9/18/18**
3881			**XU854 - Able 51 Area**	**Fri 9/20/13**	**Wed 11/7/18**
4015			**FI857 - Former Bunker**	**Fri 9/20/13**	**Wed 8/15/18**
4122			**SR858 - Former Small Arms Range**	**Tue 10/1/13**	**Mon 2/12/18**
4269			**SR859 Former Skeet Range**	**Tue 10/1/13**	**Thu 12/12/19**
4270	**SR859**	**0068AA**	**Achieve Final Remedial Investigation (RI) Report**	**Tue 10/1/13**	**Thu 1/21/16**
4271	SR859	0068AA	NTP (SR859/0068AA)	Tue 10/1/13	Tue 10/1/13
4272	SR859	0068AA	POP Completion Date	Thu 1/21/16	Thu 1/21/16
4273	**SR859**	**0068AA**	**Explosive Safety Submission (ESS)**	**Tue 5/26/15**	**Fri 1/8/16**
4274	SR859	0068AA	Prepare and Submit Draft ESS	Tue 5/26/15	Mon 6/22/15
4275	SR859	0068AA	Air Force Approval Draft ESS Milestone	Mon 6/29/15	Mon 6/29/15
4276	SR859	0068AA	Air Force Review of Draft/Provide Comments	Tue 6/23/15	Wed 7/22/15
4277	SR859	0068AA	Respond to Air Force Comments/Prepare and Submit Draft Final ESS	Thu 7/23/15	Wed 8/19/15
4278	SR859	0068AA	Air Force Review of Draft Final ESS/Revise as needed	Thu 8/20/15	Wed 9/2/15
4279	SR859	0068AA	Air Force Approval Draft Final ESS Milestone	Wed 9/2/15	Wed 9/2/15
4280	SR859	0068AA	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Review of Draft Final/Provide Comments	Thu 9/3/15	Wed 11/25/15
4281	SR859	0068AA	Respond to AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Comments on Draft Final and Prepare Final ESS	Thu 11/26/15	Wed 12/2/15
4282	SR859	0068AA	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Review of Final ESS/Revise as needed	Thu 12/3/15	Fri 1/1/16
4283	SR859	0068AA	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Approval of Final ESS Milestone	Fri 1/8/16	Fri 1/8/16
4284	**SR859**	**0068AA**	**Remedial Investigation Work Plan**	**Thu 10/17/13**	**Fri 2/13/15**
4285	SR859	0068AA	Prepare and Submit Draft Remedial Investigation Work Plan	Thu 10/17/13	Wed 11/13/13
4286	SR859	0068AA	Air Force Approval Draft Remedial Investigation Work Plan Milestone	Wed 11/20/13	Wed 11/20/13
4287	SR859	0068AA	Air Force Review of Draft/Provide Comments	Thu 11/14/13	Mon 12/9/13
4288	SR859	0068AA	Respond to Air Force Comments	Tue 12/10/13	Fri 1/10/14
4289	SR859	0068AA	Prepare and Submit Draft Final Remedial Investigation Work Plan	Mon 1/13/14	Tue 5/27/14
4290	SR859	0068AA	Air Force Approval of Draft Final Remedial Investigation Work Plan Milesto	Wed 5/28/14	Mon 6/30/14
4291	SR859	0068AA	Submit Draft Final for EPA Review	Mon 6/30/14	Mon 6/30/14
4292	SR859	0068AA	EPA Review of Draft Final/Provide Comments	Tue 7/1/14	Fri 12/5/14
4293	SR859	0068AA	Respond to EPA Comments on Draft Final and Prepare Final Remedial Investigation Work Plan	Mon 12/8/14	Wed 12/17/14
4294	SR859	0068AA	Air Force and EPA Review of Final Remedial Investigation Work Plan/Rev	Thu 12/18/14	Fri 2/13/15
4295	SR859	0068AA	EPA and Air Force Approval of Final Remedial Investigation Work Plan Milestone	Fri 2/13/15	Fri 2/13/15
4296	**SR859**	**0068AA**	**Remedial Investigation Field Activities**	**Mon 2/16/15**	**Mon 7/27/15**
4297	SR859	0068AA	UIC Permit/Utility Locates/Dig Permits/Mobilization	Mon 2/16/15	Mon 4/6/15
4298	SR859	0068AA	MEC Survey and Soil Sampling	Tue 4/7/15	Mon 4/27/15
4299	SR859	0068AA	ERPIMS Submission	Mon 7/27/15	Mon 7/27/15
4300	**SR859**	**0068AA**	**Remedial Investigation Report**	**Tue 5/26/15**	**Thu 1/21/16**
4301	SR859	0068AA	Prepare and Submit Draft Remedial Investigation Report	Tue 5/26/15	Mon 6/15/15
4302	SR859	0068AA	Air Force Approval Draft Remedial Investigation Report Milestone	Mon 6/22/15	Mon 6/22/15
4303	SR859	0068AA	Air Force Review of Draft/Provide Comments	Tue 6/16/15	Wed 7/15/15
4304	SR859	0068AA	Respond to Air Force Comments	Thu 7/16/15	Wed 7/29/15
4305	SR859	0068AA	Prepare and Submit Draft Final Remedial Investigation Report	Thu 7/30/15	Wed 8/5/15
4306	SR859	0068AA	Air Force Approval of Draft Final Remedial Investigation Report Milestone	Wed 8/5/15	Wed 8/5/15
4307	SR859	0068AA	Submit Draft Final for EPA Review	Thu 8/6/15	Thu 8/6/15
4308	SR859	0068AA	EPA Review of Draft Final/Provide Comments	Fri 8/7/15	Thu 12/24/15
4309	SR859	0068AA	Respond to EPA Comments on Draft Final and Prepare Final Remedial Investigation Report	Fri 12/25/15	Thu 12/31/15
4310	SR859	0068AA	Air Force and EPA Review of Final Remedial Investigation Report/Revise a	Fri 1/1/16	Thu 1/21/16
4311	SR859	0068AA	EPA and Air Force Approval of Final Remedial Investigation Report Milesto	Thu 1/21/16	Thu 1/21/16
4312	**SR859**	**0068AB**	**Achieve Final Non-Time Critical Removal Action (NTCRA) Work Plan**	**Thu 4/30/15**	**Tue 8/1/17**
4313	SR859	0068AB	NTP (SR859/0068AB)	Thu 4/30/15	Thu 4/30/15
4314	SR859	0068AB	POP Completion Date	Tue 8/1/17	Tue 8/1/17

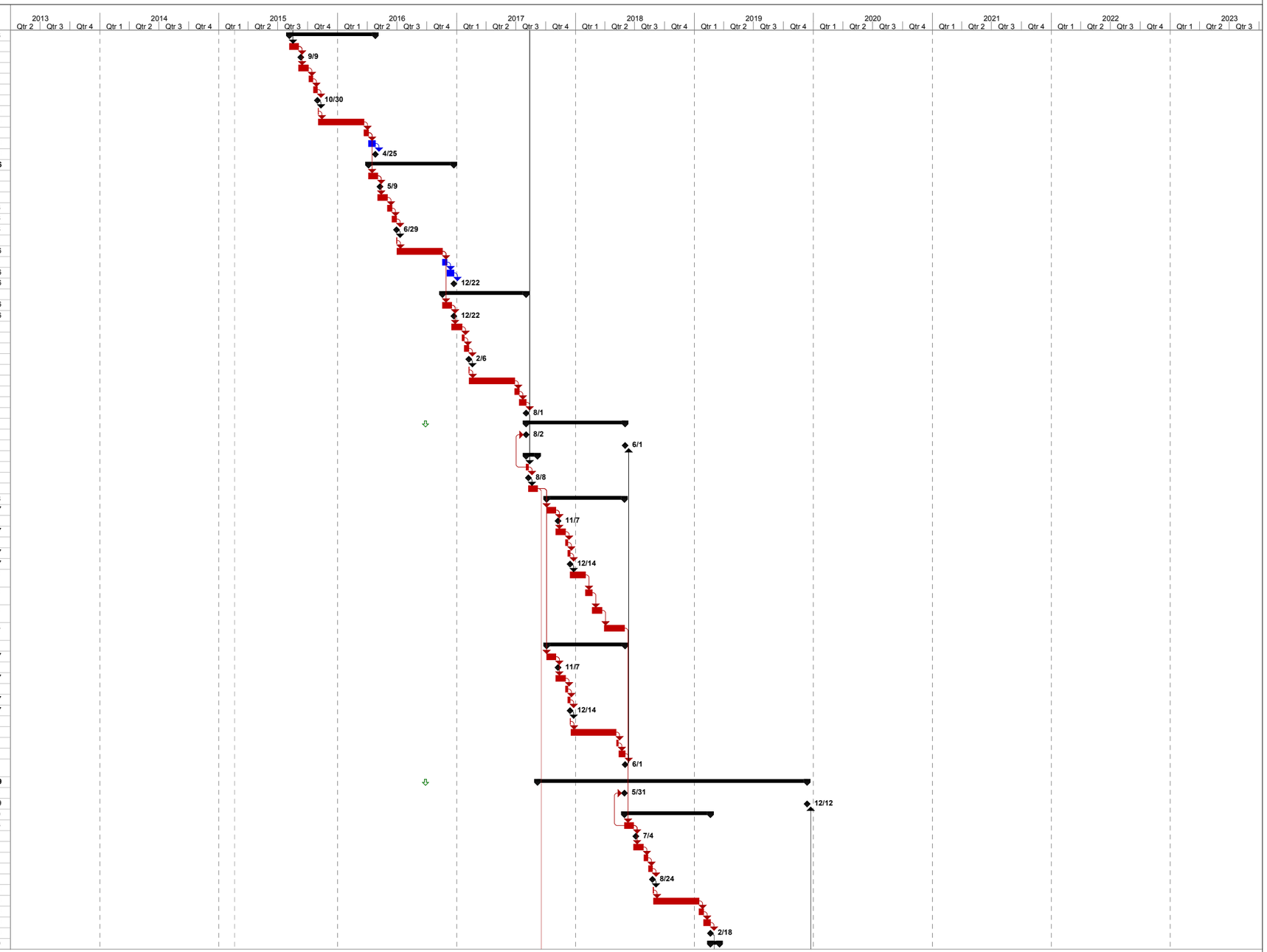


Project: Integrated Master Schedule
Date: Wed 2/18/15
File: NM-AZ Group - Holloman AFB_2-

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

New Mexico-Arizona Group
Holloman AFB, New Mexico
Performance Based Remediation
Integrated Master Schedule

ID	Site Name	SubCLIN	Name	Start	Finish
4315	**SR859**	**0068AB**	**Engineering Evaluation/Cost Analysis (EE/CA)**	**Thu 8/6/15**	**Mon 4/25/16**
4316	SR859	0068AB	Prepare and Submit Draft EE/CA	Thu 8/6/15	Wed 9/2/15
4317	SR859	0068AB	Air Force Approval Draft EE/CA Milestone	Wed 9/9/15	Wed 9/9/15
4318	SR859	0068AB	Air Force Review of Draft/Provide Comments	Thu 9/3/15	Fri 10/2/15
4319	SR859	0068AB	Respond to Air Force Comments	Mon 10/5/15	Fri 10/16/15
4320	SR859	0068AB	Prepare and Submit Draft Final EE/CA	Mon 10/19/15	Fri 10/30/15
4321	SR859	0068AB	Air Force Approval of Draft Final EE/CA Milestone	Fri 10/30/15	Fri 10/30/15
4322	SR859	0068AB	Submit Draft Final for EPA Review	Mon 11/2/15	Mon 11/2/15
4323	SR859	0068AB	EPA Review of Draft Final/Provide Comments	Tue 11/3/15	Mon 3/21/16
4324	SR859	0068AB	Respond to EPA Comments on Draft Final and Prepare Final EE/CA	Tue 3/22/16	Mon 4/4/16
4325	SR859	0068AB	Air Force and EPA Review of Final EE/CA/Revise as needed	Tue 4/5/16	Mon 4/25/16
4326	SR859	0068AB	EPA and Air Force Approval of Final EE/CA Milestone	Mon 4/25/16	Mon 4/25/16
4327	**SR859**	**0068AB**	**Action Memorandum**	**Tue 4/5/16**	**Thu 12/22/16**
4328	SR859	0068AB	Prepare and Submit Draft Action Memorandum	Tue 4/5/16	Mon 5/2/16
4329	SR859	0068AB	Air Force Approval Draft Action Memorandum Milestone	Mon 5/9/16	Mon 5/9/16
4330	SR859	0068AB	Air Force Review of Draft/Provide Comments	Tue 5/3/16	Wed 6/1/16
4331	SR859	0068AB	Respond to Air Force Comments	Thu 6/2/16	Wed 6/15/16
4332	SR859	0068AB	Prepare and Submit Draft Final Action Memorandum	Thu 6/16/16	Wed 6/29/16
4333	SR859	0068AB	Air Force Approval of Draft Final Action Memorandum Milestone	Wed 6/29/16	Wed 6/29/16
4334	SR859	0068AB	Submit Draft Final for EPA Review	Thu 6/30/16	Thu 6/30/16
4335	SR859	0068AB	EPA Review of Draft Final/Provide Comments	Fri 7/1/16	Thu 11/17/16
4336	SR859	0068AB	Respond to EPA Comments on Draft Final and Prepare Action Memorandu	Fri 11/18/16	Thu 12/1/16
4337	SR859	0068AB	Air Force and EPA Review of Final Action Memorandum/Revise as needed	Fri 12/2/16	Thu 12/22/16
4338	SR859	0068AB	EPA and Air Force Approval of Final Action Memorandum Milestone	Thu 12/22/16	Thu 12/22/16
4339	**SR859**	**0068AB**	**Non-Time Critical Removal Action (NTCRA) Work Plan**	**Fri 11/18/16**	**Tue 8/1/17**
4340	SR859	0068AB	Prepare and Submit Draft NTCRA Work Plan	Fri 11/18/16	Thu 12/15/16
4341	SR859	0068AB	Air Force Approval Draft NTCRA Work Plan Milestone	Thu 12/22/16	Thu 12/22/16
4342	SR859	0068AB	Air Force Review of Draft/Provide Comments	Fri 12/16/16	Mon 1/16/17
4343	SR859	0068AB	Respond to Air Force Comments	Tue 1/17/17	Mon 1/23/17
4344	SR859	0068AB	Prepare and Submit Draft Final NTCRA Work Plan	Tue 1/24/17	Mon 2/6/17
4345	SR859	0068AB	Air Force Approval of Draft Final NTCRA Work Plan Milestone	Mon 2/6/17	Mon 2/6/17
4346	SR859	0068AB	Submit Draft Final for EPA Review	Tue 2/7/17	Tue 2/7/17
4347	SR859	0068AB	EPA Review of Draft Final/Provide Comments	Wed 2/8/17	Tue 6/27/17
4348	SR859	0068AB	Respond to EPA Comments on Draft Final and Prepare NTCRA Work Plar	Wed 6/28/17	Tue 7/11/17
4349	SR859	0068AB	Air Force and EPA Review of Final NTCRA Work Plan/Revise as needed	Wed 7/12/17	Tue 8/1/17
4350	SR859	0068AB	EPA and Air Force Approval of Final NTCRA Work Plan Milestone	Tue 8/1/17	Tue 8/1/17
4351	**SR859**	**0068AC**	**Achieve Response Complete (RC)**	**Wed 8/2/17**	**Fri 6/1/18**
4352	SR859	0068AC	NTP (SR859/0068AC)	Wed 8/2/17	Wed 8/2/17
4353	SR859	0068AC	POP Completion Date	Fri 6/1/18	Fri 6/1/18
4354	**SR859**	**0068AC**	**NTCRA Field Activities**	**Wed 8/2/17**	**Tue 9/5/17**
4355	SR859	0068AC	UIC Permit/Utility Locates/Dig Permits/Mobilization	Wed 8/2/17	Tue 8/8/17
4356	SR859	0068AC	Construction Bond	Tue 8/8/17	Tue 8/8/17
4357	SR859	0068AC	Soil Removal, Soil Confirmatory Sampling, and Site Restoration	Wed 8/9/17	Tue 9/5/17
4358	**SR859**	**0068AC**	**Certificate of Clearance**	**Wed 10/4/17**	**Wed 5/30/18**
4359	SR859	0068AC	Prepare and Submit Draft Certificate of Clearance	Wed 10/4/17	Tue 10/31/17
4360	SR859	0068AC	Air Force Approval Draft Certificate of Clearance Milestone	Tue 11/7/17	Tue 11/7/17
4361	SR859	0068AC	Air Force Review of Draft/Provide Comments	Wed 11/1/17	Thu 11/30/17
4362	SR859	0068AC	Respond to Air Force Comments/Prepare and Submit Draft Final Certifica	Fri 12/1/17	Thu 12/7/17
4363	SR859	0068AC	Air Force Review of Draft Final Certificate of Clearance/Revise as needed	Fri 12/8/17	Thu 12/14/17
4364	SR859	0068AC	Air Force Approval Draft Final Certificate of Clearance Milestone	Thu 12/14/17	Thu 12/14/17
4365	SR859	0068AC	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Review of Draft Final/Provide Comments	Fri 12/15/17	Tue 1/30/18
4366	SR859	0068AC	Respond to AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Comments on Draft Final and Prepare Final Certificate of	Wed 1/31/18	Tue 2/20/18
4367	SR859	0068AC	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Review of Final Certificate of Clearance/Revise as needed	Wed 2/21/18	Thu 3/22/18
4368	SR859	0068AC	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Approval of Final Certificate of Clearance Milestone	Fri 3/30/18	Wed 5/30/18
4369	**SR859**	**0068AC**	**After Action Report**	**Wed 10/4/17**	**Fri 6/1/18**
4370	SR859	0068AC	Prepare and Submit Draft After Action Report	Wed 10/4/17	Tue 10/31/17
4371	SR859	0068AC	Air Force Approval Draft After Action Report Milestone	Tue 11/7/17	Tue 11/7/17
4372	SR859	0068AC	Air Force Review of Draft/Provide Comments	Wed 11/1/17	Thu 11/30/17
4373	SR859	0068AC	Respond to Air Force Comments	Fri 12/1/17	Thu 12/7/17
4374	SR859	0068AC	Prepare and Submit Draft Final After Action Report	Fri 12/8/17	Thu 12/14/17
4375	SR859	0068AC	Air Force Approval of Draft Final After Action Report Milestone	Thu 12/14/17	Thu 12/14/17
4376	SR859	0068AC	Submit Draft Final for EPA Review	Fri 12/15/17	Fri 12/15/17
4377	SR859	0068AC	EPA Review of Draft Final/Provide Comments	Mon 12/18/17	Fri 5/4/18
4378	SR859	0068AC	Respond to EPA Comments on Draft Final and Prepare After Action Repo	Mon 5/7/18	Fri 5/11/18
4379	SR859	0068AC	Air Force and EPA Review of Final After Action Report/Revise as needed	Mon 5/14/18	Fri 6/1/18
4380	SR859	0068AC	EPA and Air Force Approval of Final After Action Report Milestone (Achieve RC)	Fri 6/1/18	Fri 6/1/18
4381	**SR859**	**0068AD**	**Achieve Site Closeout (SC) in accordance with Table 1 of the SOO**	**Wed 9/6/17**	**Thu 12/12/19**
4382	SR859	0068AD	NTP (SR859/0068AA)	Thu 5/31/18	Thu 5/31/18
4383	SR859	0068AD	POP Completion Date	Thu 12/12/19	Thu 12/12/19
4384	**SR859**	**0068AD**	**Proposed Plan**	**Thu 5/31/18**	**Mon 2/18/19**
4385	SR859	0068AD	Prepare and Submit Draft Proposed Plan	Thu 5/31/18	Wed 6/27/18
4386	SR859	0068AD	Air Force Approval Draft Proposed Plan Milestone	Wed 7/4/18	Wed 7/4/18
4387	SR859	0068AD	Air Force Review of Draft/Provide Comments	Thu 6/28/18	Fri 7/27/18
4388	SR859	0068AD	Respond to Air Force Comments	Mon 7/30/18	Fri 8/10/18
4389	SR859	0068AD	Prepare and Submit Draft Final Proposed Plan	Mon 8/13/18	Fri 8/24/18
4390	SR859	0068AD	Air Force Approval of Draft Final Proposed Plan Milestone	Fri 8/24/18	Fri 8/24/18
4391	SR859	0068AD	Submit Draft Final for EPA Review	Mon 8/27/18	Mon 8/27/18
4392	SR859	0068AD	EPA Review of Draft Final/Provide Comments	Tue 8/28/18	Mon 1/14/19
4393	SR859	0068AD	Respond to EPA Comments on Draft Final and Prepare Final Proposed Pl	Tue 1/15/19	Mon 1/28/19
4394	SR859	0068AD	Air Force and EPA Review of Final Proposed Plan /Revise as needed	Tue 1/29/19	Mon 2/18/19
4395	SR859	0068AD	EPA and Air Force Approval of Final Proposed Plan Milestone	Mon 2/18/19	Mon 2/18/19
4396	**SR859**	**0068AD**	**Public Comment Period**	**Tue 2/19/19**	**Mon 3/18/19**



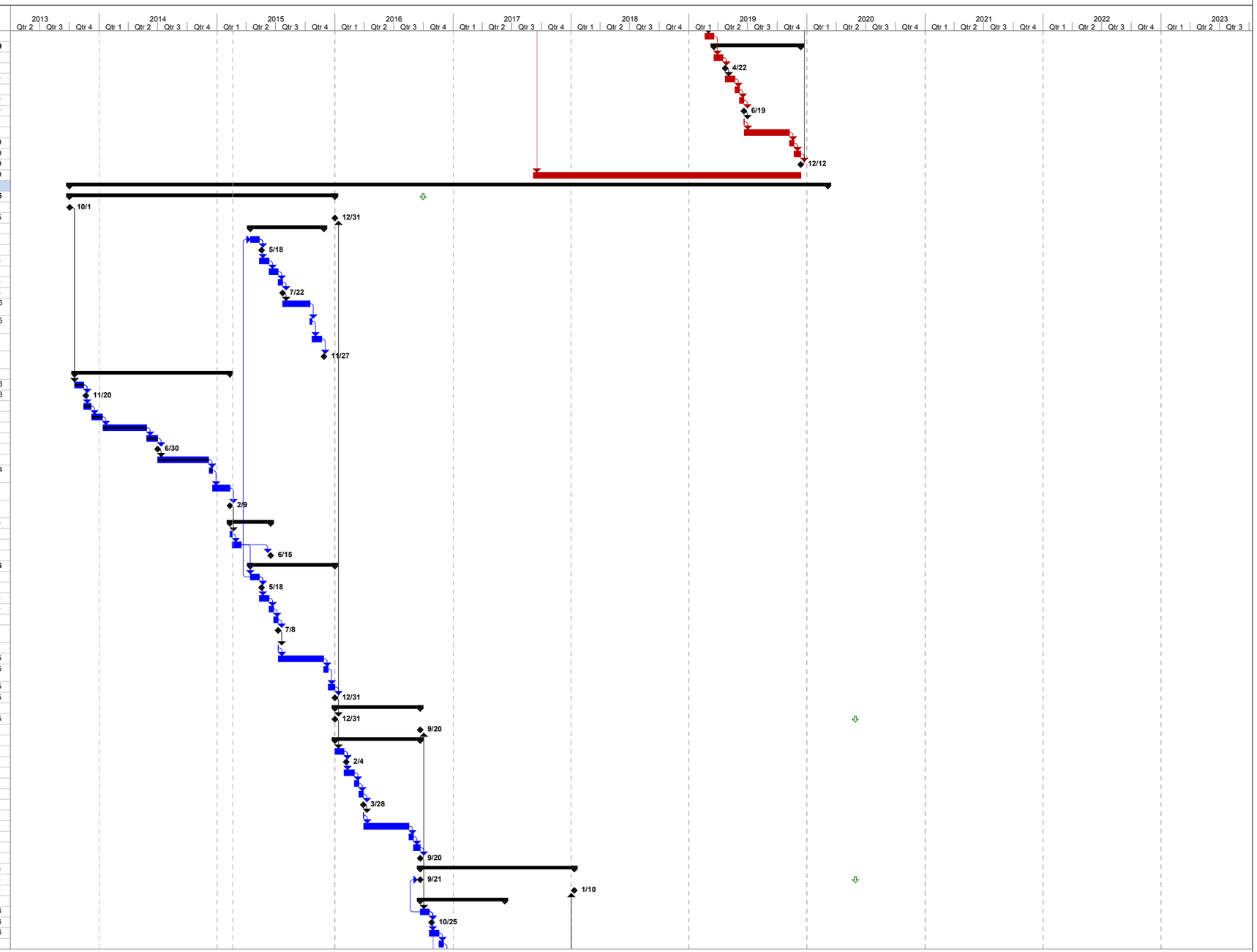
Project: Integrated Master Schedule
Date: Wed 2/18/15
File: NM-AZ Group - Holloman AFB_2-

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

New Mexico-Arizona Group
Holloman AFB, New Mexico
Performance Based Remediation
Integrated Master Schedule

FPM Remediations, Inc.

ID	Site Name	SubCLIN	Name	Start	Finish
4397	SR859	0068AD	Comment Period	Tue 2/19/19	Mon 3/18/19
4398	**SR859**	**0068AD**	**No Further Action Record of Decision (NFA ROD)**	**Tue 3/19/19**	**Thu 12/12/19**
4399	SR859	0068AD	Prepare and Submit Draft NFA ROD	Tue 3/19/19	Mon 4/15/19
4400	SR859	0068AD	Air Force Approval Draft NFA ROD Milestone	Mon 4/22/19	Mon 4/22/19
4401	SR859	0068AD	Air Force Review of Draft/Provide Comments	Tue 4/23/19	Wed 5/22/19
4402	SR859	0068AD	Respond to Air Force Comments	Thu 5/23/19	Wed 6/5/19
4403	SR859	0068AD	Prepare and Submit Draft Final NFA ROD	Thu 6/6/19	Wed 6/19/19
4404	SR859	0068AD	Air Force Approval of Draft Final NFA ROD Milestone	Wed 6/19/19	Wed 6/19/19
4405	SR859	0068AD	Submit Draft Final for EPA Review	Thu 6/20/19	Thu 6/20/19
4406	SR859	0068AD	EPA Review of Draft Final/Provide Comments	Fri 6/21/19	Thu 11/7/19
4407	SR859	0068AD	Respond to EPA Comments on Draft Final and Prepare Final NFA ROD	Fri 11/8/19	Thu 11/21/19
4408	SR859	0068AD	Air Force and EPA Review of Final NFA ROD/Revise as needed	Fri 11/22/19	Thu 12/12/19
4409	SR859	0068AD	EPA and Air Force Approval of Final NFA ROD Milestone	Thu 12/12/19	Thu 12/12/19
4410	SR859	0068AD	PBR Contractor Plan B (Trigger Evaluation/Implementation)	Wed 9/6/17	Thu 12/12/19
4411			**TS862 - Jeep Target Area Skeet Range**	**Tue 10/1/13**	**Thu 3/5/20**
4412	**TS862**	**0069AA**	**Achieve Final Remedial Investigation (RI) Report**	**Tue 10/1/13**	**Thu 12/31/15**
4413	TS862	0069AA	NTP (TS862/0069AA)	Tue 10/1/13	Tue 10/1/13
4414	TS862	0069AA	POP Completion Date	Thu 12/31/15	Thu 12/31/15
4415	**TS862**	**0069AA**	**Explosive Safety Submission (ESS)**	**Tue 4/14/15**	**Fri 11/27/15**
4416	TS862	0069AA	Prepare and Submit Draft ESS	Tue 4/14/15	Mon 5/11/15
4417	TS862	0069AA	Air Force Approval Draft ESS Milestone	Mon 5/18/15	Mon 5/18/15
4418	TS862	0069AA	Air Force Review of Draft/Provide Comments	Tue 5/12/15	Wed 6/10/15
4419	TS862	0069AA	Respond to Air Force Comments/Prepare and Submit Draft Final ESS	Thu 6/11/15	Wed 7/8/15
4420	TS862	0069AA	Air Force Review of Draft Final ESS/Revise as needed	Thu 7/9/15	Wed 7/22/15
4421	TS862	0069AA	Air Force Approval Draft Final ESS Milestone	Wed 7/22/15	Wed 7/22/15
4422	TS862	0069AA	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Review of Draft Final/Provide Comments	Thu 7/23/15	Wed 10/14/15
4423	TS862	0069AA	Respond to AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Comments on Draft Final and Prepare Final ESS	Thu 10/15/15	Wed 10/21/15
4424	TS862	0069AA	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Review of Final ESS/Revise as needed	Thu 10/22/15	Fri 11/20/15
4425	TS862	0069AA	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Approval of Final ESS Milestone	Fri 11/27/15	Fri 11/27/15
4426	**TS862**	**0069AA**	**Remedial Investigation Work Plan**	**Thu 10/17/13**	**Mon 2/9/15**
4427	TS862	0069AA	Prepare and Submit Draft Remedial Investigation Work Plan	Thu 10/17/13	Wed 11/13/13
4428	TS862	0069AA	Air Force Approval Draft Remedial Investigation Work Plan Milestone	Wed 11/20/13	Wed 11/20/13
4429	TS862	0069AA	Air Force Review of Draft/Provide Comments	Thu 11/14/13	Fri 12/6/13
4430	TS862	0069AA	Respond to Air Force Comments	Mon 12/9/13	Fri 1/10/14
4431	TS862	0069AA	Prepare and Submit Draft Final Remedial Investigation Work Plan	Mon 1/13/14	Tue 5/27/14
4432	TS862	0069AA	Air Force Approval of Draft Final Remedial Investigation Work Plan Milestc	Wed 5/28/14	Mon 6/30/14
4433	TS862	0069AA	Submit Draft Final for EPA Review	Mon 6/30/14	Mon 6/30/14
4434	TS862	0069AA	EPA Review of Draft Final/Provide Comments	Tue 7/1/14	Fri 12/5/14
4435	TS862	0069AA	Respond to EPA Comments on Draft Final and Prepare Final Remedial Investigation Work Plan	Mon 12/8/14	Wed 12/17/14
4436	TS862	0069AA	Air Force and EPA Review of Final Remedial Investigation Work Plan/Revise as needed	Thu 12/18/14	Mon 2/9/15
4437	TS862	0069AA	EPA and Air Force Approval of Final Remedial Investigation Work Plan Milestone	Mon 2/9/15	Mon 2/9/15
4438	**TS862**	**0069AA**	**Remedial Investigation Field Activities**	**Tue 2/10/15**	**Mon 6/15/15**
4439	TS862	0069AA	UIC Permit/Utility Locates/Dig Permits/Mobilization	Tue 2/10/15	Mon 2/16/15
4440	TS862	0069AA	MEC Survey and Soil Sampling	Tue 2/17/15	Mon 3/16/15
4441	TS862	0069AA	ERPIMS Submission	Mon 6/15/15	Mon 6/15/15
4442	**TS862**	**0069AA**	**Remedial Investigation Report**	**Tue 4/14/15**	**Thu 12/31/15**
4443	TS862	0069AA	Prepare and Submit Draft Remedial Investigation Report	Tue 4/14/15	Mon 5/11/15
4444	TS862	0069AA	Air Force Approval Draft Remedial Investigation Report Milestone	Mon 5/18/15	Mon 5/18/15
4445	TS862	0069AA	Air Force Review of Draft/Provide Comments	Tue 5/12/15	Wed 6/10/15
4446	TS862	0069AA	Respond to Air Force Comments	Thu 6/11/15	Wed 6/24/15
4447	TS862	0069AA	Prepare and Submit Draft Final Remedial Investigation Report	Thu 6/25/15	Wed 7/8/15
4448	TS862	0069AA	Air Force Approval of Draft Final Remedial Investigation Report Milestone	Wed 7/8/15	Wed 7/8/15
4449	TS862	0069AA	Submit Draft Final for EPA Review	Thu 7/9/15	Thu 7/9/15
4450	TS862	0069AA	EPA Review of Draft Final/Provide Comments	Fri 7/10/15	Thu 11/26/15
4451	TS862	0069AA	Respond to EPA Comments on Draft Final and Prepare Final Remedial Investigation Report	Fri 11/27/15	Thu 12/10/15
4452	TS862	0069AA	Air Force and EPA Review of Final Remedial Investigation Report/Revise a	Fri 12/11/15	Thu 12/31/15
4453	TS862	0069AA	EPA and Air Force Approval of Final Remedial Investigation Report Milest	Thu 12/31/15	Thu 12/31/15
4454	**TS862**	**0069AB**	**Achieve Final Engineering Evaluatin/Cost Analysis (EE/CA)**	**Thu 12/31/15**	**Tue 9/20/16**
4455	TS862	0069AB	NTP (TS862/0069AB)	Thu 12/31/15	Thu 12/31/15
4456	TS862	0069AB	POP Completion Date	Tue 9/20/16	Tue 9/20/16
4457	**TS862**	**0069AB**	**Engineering Evaluation/Cost Analysis (EE/CA)**	**Fri 1/1/16**	**Tue 9/20/16**
4458	TS862	0069AB	Prepare and Submit Draft EE/CA	Fri 1/1/16	Thu 1/28/16
4459	TS862	0069AB	Air Force Approval Draft EE/CA Milestone	Thu 2/4/16	Thu 2/4/16
4460	TS862	0069AB	Air Force Review of Draft/Provide Comments	Fri 1/29/16	Mon 2/29/16
4461	TS862	0069AB	Respond to Air Force Comments	Tue 3/1/16	Mon 3/14/16
4462	TS862	0069AB	Prepare and Submit Draft Final EE/CA	Tue 3/15/16	Mon 3/28/16
4463	TS862	0069AB	Air Force Approval of Draft Final EE/CA Milestone	Mon 3/28/16	Mon 3/28/16
4464	TS862	0069AB	Submit Draft Final for EPA Review	Tue 3/29/16	Tue 3/29/16
4465	TS862	0069AB	EPA Review of Draft Final/Provide Comments	Wed 3/30/16	Tue 8/16/16
4466	TS862	0069AB	Respond to EPA Comments on Draft Final and Prepare Final EE/CA	Wed 8/17/16	Tue 8/30/16
4467	TS862	0069AB	Air Force and EPA Review of Final EE/CA/Revise as needed	Wed 8/31/16	Tue 9/20/16
4468	TS862	0069AB	EPA and Air Force Approval of Final EE/CA Milestone	Tue 9/20/16	Tue 9/20/16
4469	**TS862**	**0069AC**	**Achieve Final Non-Time Critical Removal Action (NTCRA) Work Plan**	**Wed 9/21/16**	**Wed 1/10/18**
4470	TS862	0069AC	NTP (TS862/0069AC)	Wed 9/21/16	Wed 9/21/16
4471	TS862	0069AC	POP Completion Date	Wed 1/10/18	Wed 1/10/18
4472	**TS862**	**0069AC**	**Action Memorandum**	**Wed 9/21/16**	**Fri 6/9/17**
4473	TS862	0069AC	Prepare and Submit Draft Action Memorandum	Wed 9/21/16	Tue 10/18/16
4474	TS862	0069AC	Air Force Approval Draft Action Memorandum Milestone	Tue 10/25/16	Tue 10/25/16
4475	TS862	0069AC	Air Force Review of Draft/Provide Comments	Wed 10/19/16	Thu 11/17/16
4476	TS862	0069AC	Respond to Air Force Comments	Fri 11/18/16	Thu 12/1/16



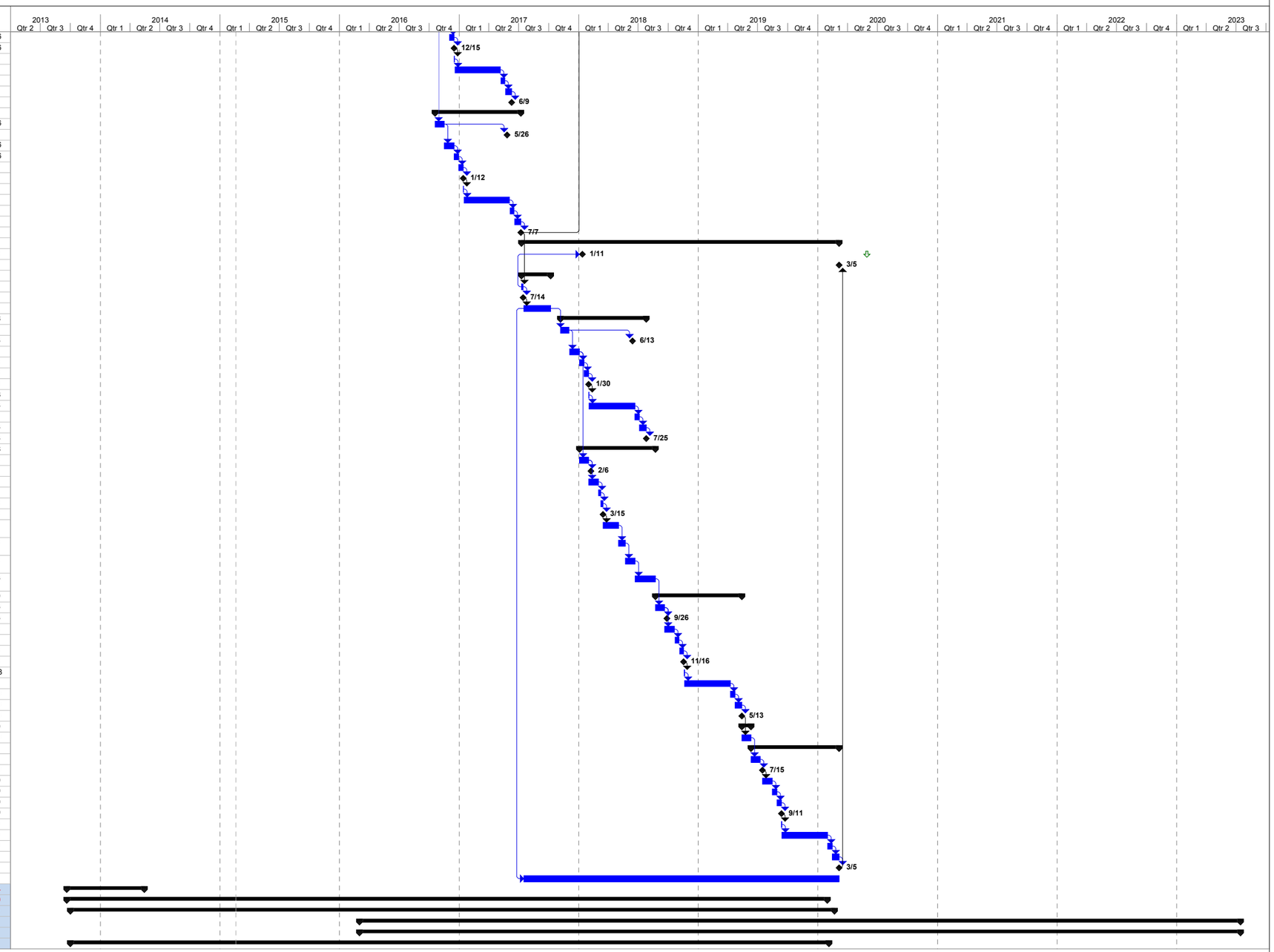
Project: Integrated Master Schedule
Date: Wed 2/18/15
File: NM-AZ Group - Holloman AFB_2-

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

New Mexico-Arizona Group
Holloman AFB, New Mexico
Performance Based Remediation
Integrated Master Schedule

FPM Remediations, Inc.

ID	Site Name	SubCLIN	Name	Start	Finish
4477	TS862	0069AC	Prepare and Submit Draft Final Action Memorandum	Fri 12/2/16	Thu 12/15/16
4478	TS862	0069AC	Air Force Approval of Draft Final Action Memorandum Milestone	Thu 12/15/16	Thu 12/15/16
4479	TS862	0069AC	Submit Draft Final for EPA Review	Fri 12/16/16	Fri 12/16/16
4480	TS862	0069AC	EPA Review of Draft Final/Provide Comments	Mon 12/19/16	Fri 5/5/17
4481	TS862	0069AC	Respond to EPA Comments on Draft Final and Prepare Final Action Memo	Mon 5/8/17	Fri 5/19/17
4482	TS862	0069AC	Air Force and EPA Review of Final Action Memorandum/Revise as needed	Mon 5/22/17	Fri 6/9/17
4483	TS862	0069AC	EPA and Air Force Approval of Final Action Memorandum Milestone	Fri 6/9/17	Fri 6/9/17
4484	**TS862**	**0069AC**	**NTCRA Work Plan**	**Wed 10/19/16**	**Fri 7/7/17**
4485	TS862	0069AC	Prepare and Submit Draft NTCRA Work Plan	Wed 10/19/16	Tue 11/15/16
4486	TS862	0069AC	Air Force Approval Draft NTCRA Work Plan Milestone	Fri 5/26/17	Fri 5/26/17
4487	TS862	0069AC	Air Force Review of Draft/Provide Comments	Wed 11/16/16	Thu 12/15/16
4488	TS862	0069AC	Respond to Air Force Comments	Fri 12/16/16	Thu 12/29/16
4489	TS862	0069AC	Prepare and Submit Draft Final NTCRA Work Plan	Fri 12/30/16	Thu 1/12/17
4490	TS862	0069AC	Air Force Approval of Draft Final NTCRA Work Plan Milestone	Thu 1/12/17	Thu 1/12/17
4491	TS862	0069AC	Submit Draft Final for EPA Review	Fri 1/13/17	Fri 1/13/17
4492	TS862	0069AC	EPA Review of Draft Final/Provide Comments	Mon 1/16/17	Fri 6/2/17
4493	TS862	0069AC	Respond to EPA Comments on Draft Final and Prepare Final NTCRA Wor	Mon 6/5/17	Fri 6/16/17
4494	TS862	0069AC	Air Force and EPA Review of Final NTCRA Work Plan/Revise as needed	Mon 6/19/17	Fri 7/7/17
4495	TS862	0069AC	EPA and Air Force Approval of Final NTCRA Work Plan Milestone	Fri 7/7/17	Fri 7/7/17
4496	**TS862**	**0069AD**	**Achieve Site Closeout (SC) in accordance with Table 1 of the SOO**	**Mon 7/10/17**	**Thu 3/5/20**
4497	TS862	0069AD	NTP (TS862/0069AD)	Thu 1/11/18	Thu 1/11/18
4498	TS862	0069AD	POP Completion Date	Thu 3/5/20	Thu 3/5/20
4499	**TS862**	**0069AD**	**NTCRA Field Activities**	**Mon 7/10/17**	**Fri 10/6/17**
4500	TS862	0069AD	UIC Permit/Utility Locates/Dig Permits/Mobilization	Mon 7/10/17	Fri 7/14/17
4501	TS862	0069AD	Construction Bond	Fri 7/14/17	Fri 7/14/17
4502	TS862	0069AD	Soil Removal, Soil Confirmatory Sampling, and Site Restoration	Mon 7/17/17	Fri 10/6/17
4503	**TS862**	**0069AD**	**After Action Report**	**Mon 11/6/17**	**Wed 7/25/18**
4504	TS862	0069AD	Prepare and Submit Draft After Action Report	Mon 11/6/17	Fri 12/1/17
4505	TS862	0069AD	Air Force Approval Draft After Action Report Milestone	Wed 6/13/18	Wed 6/13/18
4506	TS862	0069AD	Air Force Review of Draft/Provide Comments	Mon 12/4/17	Tue 1/2/18
4507	TS862	0069AD	Respond to Air Force Comments	Wed 1/3/18	Tue 1/16/18
4508	TS862	0069AD	Prepare and Submit Draft Final After Action Report	Wed 1/17/18	Tue 1/30/18
4509	TS862	0069AD	Air Force Approval of Draft Final After Action Report Milestone	Tue 1/30/18	Tue 1/30/18
4510	TS862	0069AD	Submit Draft Final for EPA Review	Wed 1/31/18	Wed 1/31/18
4511	TS862	0069AD	EPA Review of Draft Final/Provide Comments	Thu 2/1/18	Wed 6/20/18
4512	TS862	0069AD	Respond to EPA Comments on Draft Final and Prepare Final After Action	Thu 6/21/18	Wed 7/4/18
4513	TS862	0069AD	Air Force and EPA Review of Final After Action Report/Revise as needed	Thu 7/5/18	Wed 7/25/18
4514	TS862	0069AD	EPA and Air Force Approval of Final After Action Report Milestone	Wed 7/25/18	Wed 7/25/18
4515	**TS862**	**0069AD**	**Certificate of Clearance**	**Wed 1/3/18**	**Wed 8/22/18**
4516	TS862	0069AD	Prepare and Submit Draft Certificate of Clearance	Wed 1/3/18	Tue 1/30/18
4517	TS862	0069AD	Air Force Approval Draft Certificate of Clearance Milestone	Tue 2/6/18	Tue 2/6/18
4518	TS862	0069AD	Air Force Review of Draft/Provide Comments	Wed 1/31/18	Thu 3/1/18
4519	TS862	0069AD	Respond to Air Force Comments/Prepare and Submit Draft Final Certificat	Fri 3/2/18	Thu 3/8/18
4520	TS862	0069AD	Air Force Review of Draft Final Certificate of Clearance/Revise as needed	Fri 3/9/18	Thu 3/15/18
4521	TS862	0069AD	Air Force Approval Draft Final Certificate of Clearance Milestone	Thu 3/15/18	Thu 3/15/18
4522	TS862	0069AD	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Review of Draft Final/Provide Comments	Fri 3/16/18	Tue 5/1/18
4523	TS862	0069AD	Respond to AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Comments on Draft Final and Prepare Final Certificate of	Wed 5/2/18	Tue 5/22/18
4524	TS862	0069AD	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Review of Final Certificate of Clearance/Revise as needed	Wed 5/23/18	Thu 6/21/18
4525	TS862	0069AD	AFCEC, Wing Safety, Command Safety, AF Safety Center, and DDESB Approval of Final Certificate of Clearance Milestone	Fri 6/22/18	Wed 8/22/18
4526	**TS862**	**0069AD**	**Proposed Plan**	**Thu 8/23/18**	**Mon 5/13/19**
4527	TS862	0069AD	Prepare and Submit Draft Proposed Plan	Thu 8/23/18	Wed 9/19/18
4528	TS862	0069AD	Air Force Approval Draft Proposed Plan Milestone	Wed 9/26/18	Wed 9/26/18
4529	TS862	0069AD	Air Force Review of Draft/Provide Comments	Thu 9/20/18	Fri 10/19/18
4530	TS862	0069AD	Respond to Air Force Comments	Mon 10/22/18	Fri 11/2/18
4531	TS862	0069AD	Prepare and Submit Draft Final Proposed Plan	Mon 11/5/18	Fri 11/16/18
4532	TS862	0069AD	Air Force Approval of Draft Final Proposed Plan Milestone	Fri 11/16/18	Fri 11/16/18
4533	TS862	0069AD	Submit Draft Final for EPA Review	Mon 11/19/18	Mon 11/19/18
4534	TS862	0069AD	EPA Review of Draft Final/Provide Comments	Tue 11/20/18	Mon 4/8/19
4535	TS862	0069AD	Respond to EPA Comments on Draft Final and Prepare Final Proposed Pl	Tue 4/9/19	Mon 4/22/19
4536	TS862	0069AD	Air Force and EPA Review of Final Proposed Plan /Revise as needed	Tue 4/23/19	Mon 5/13/19
4537	TS862	0069AD	EPA and Air Force Approval of Final Proposed Plan Milestone	Mon 5/13/19	Mon 5/13/19
4538	**TS862**	**0069AD**	**Public Comment Period**	**Tue 5/14/19**	**Mon 6/10/19**
4539	TS862	0069AD	Comment Period	Tue 5/14/19	Mon 6/10/19
4540	**TS862**	**0069AD**	**No Further Action Record of Decision (NFA ROD)**	**Tue 6/11/19**	**Thu 3/5/20**
4541	TS862	0069AD	Prepare and Submit Draft NFA ROD	Tue 6/11/19	Mon 7/8/19
4542	TS862	0069AD	Air Force Approval Draft NFA ROD Milestone	Mon 7/15/19	Mon 7/15/19
4543	TS862	0069AD	Air Force Review of Draft/Provide Comments	Tue 7/16/19	Wed 8/14/19
4544	TS862	0069AD	Respond to Air Force Comments	Thu 8/15/19	Wed 8/28/19
4545	TS862	0069AD	Prepare and Submit Draft Final NFA ROD	Thu 8/29/19	Wed 9/11/19
4546	TS862	0069AD	Air Force Approval of Draft Final NFA ROD Milestone	Wed 9/11/19	Wed 9/11/19
4547	TS862	0069AD	Submit Draft Final for EPA Review	Thu 9/12/19	Thu 9/12/19
4548	TS862	0069AD	EPA Review of Draft Final/Provide Comments	Fri 9/13/19	Thu 1/30/20
4549	TS862	0069AD	Respond to EPA Comments on Draft Final and Prepare Final NFA ROD	Fri 1/31/20	Thu 2/13/20
4550	TS862	0069AD	Air Force and EPA Review of Final NFA ROD /Revise as needed	Fri 2/14/20	Thu 3/5/20
4551	TS862	0069AD	EPA and Air Force Approval of Final NFA ROD Milestone	Thu 3/5/20	Thu 3/5/20
4552	TS862	0069AD	PBR Contractor Plan B (Trigger Evaluation/Implementation)	Mon 7/17/17	Thu 3/5/20
4553			**SR863 - 1000 Inch Range**	**Fri 9/20/13**	**Wed 5/14/14**
4595			**SR864 - Poorman Range**	**Fri 9/20/13**	**Wed 1/29/20**
4733			**ML865 - Ballistics Rain Field**	**Tue 10/1/13**	**Thu 2/20/20**
4871			**ML866 - Former Bombing Range**	**Tue 3/1/16**	**Fri 7/14/23**
5013			**SR867 - Possible Firing Range**	**Tue 3/1/16**	**Fri 7/14/23**
5155			**RR869 - Debris Field**	**Tue 10/1/13**	**Mon 2/3/20**



Project: Integrated Master Schedule
Date: Wed 2/18/15
File: NM-AZ Group - Holloman AFB_2-

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

APPENDIX D
Health and Safety Activity Hazard Analysis

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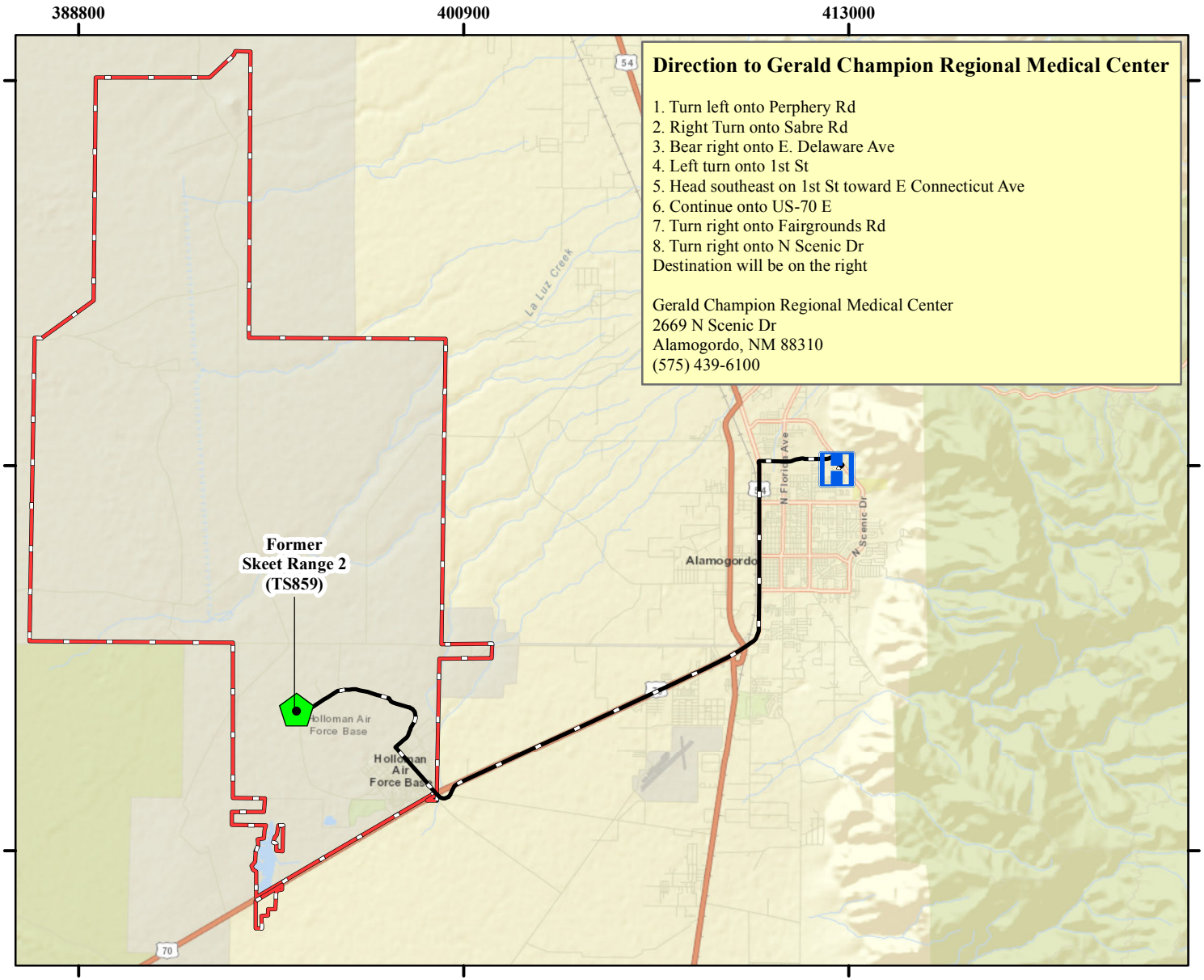
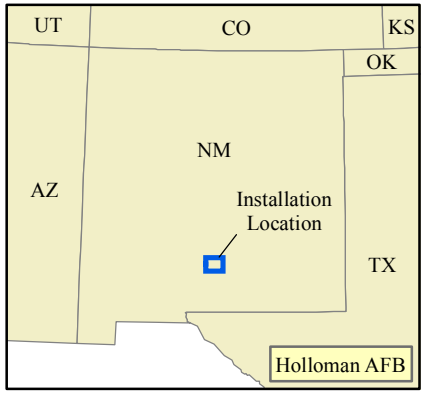




Legend

- Hospital
- SR859
- Hospital Route
- Installation Boundary

Holloman
Air Force Base

Air Force Civil Engineer Center
2261 Huges Ave., Suite 163
Lackland AFB, Texas 78236-9853

Hospital Route

Former Skeet Range 2
(SR859)

FPM Remediations, Inc.

2013

NOTES:
Revision Date: 11/12/2013

Coordinate System:NAD 1983 UTM Zone 13N
Projection: Transverse Mercator
Horizontal Datum: North American 1983
False Easting: 500,000.0000
False Northing: 0.0000
Central Meridian: -105.0000
Scale Factor: 0.9996
Latitude Of Origin: 0.0000
Units: Meter

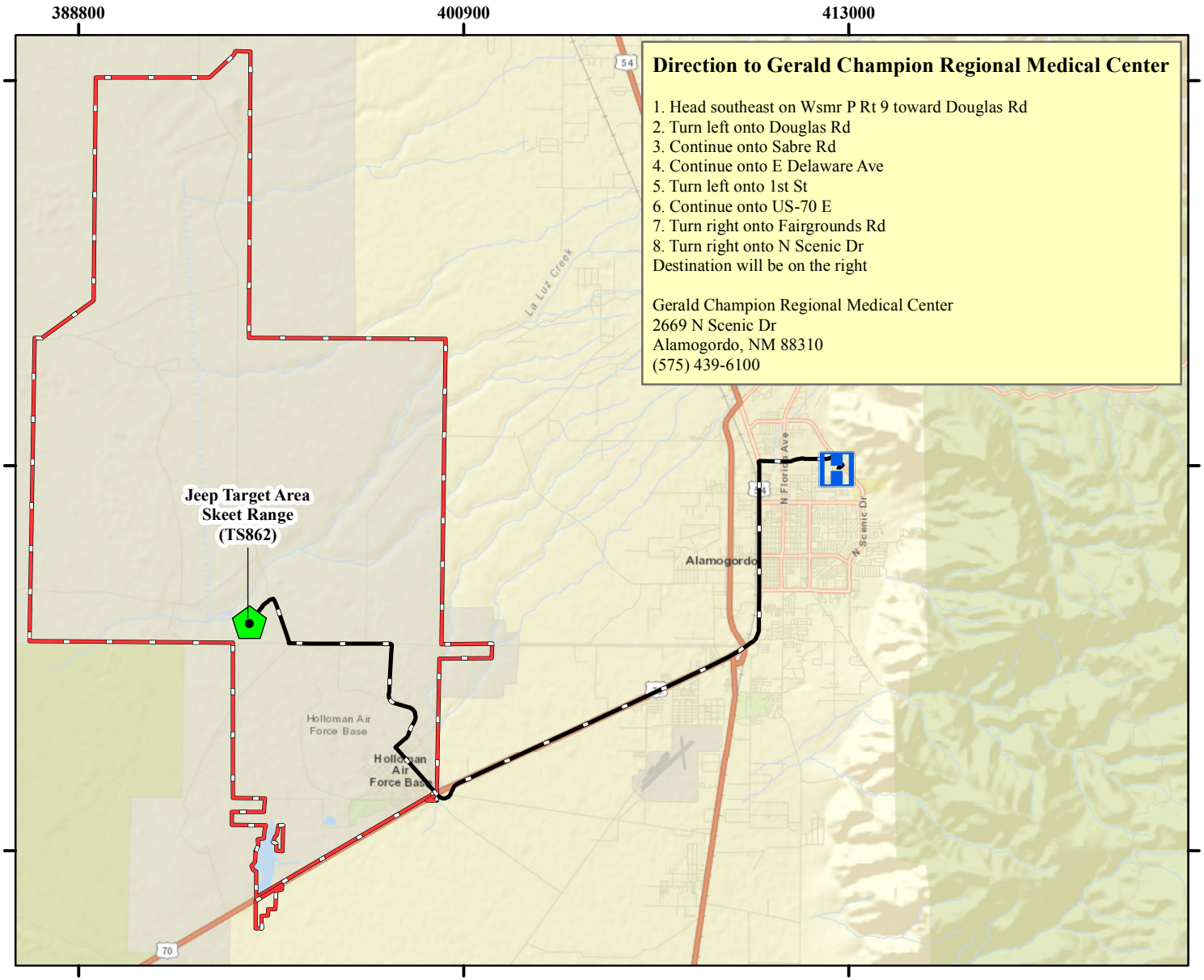
1 inch = 16,000 feet

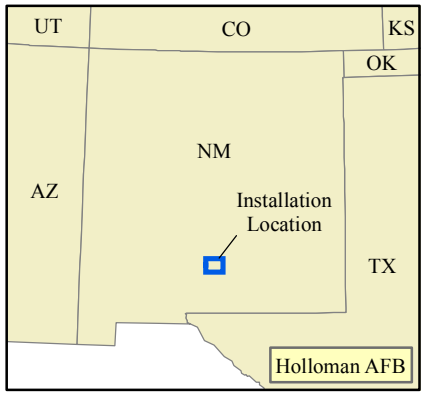
0 2.5 5 Kilometers

0 1.5 3 Miles

N W E S

Path: C:\GIS\Projects\Holloman_AFB\Projects\Hospital_Routes\SR859_Hospital_Route.mxd





Legend

- Hospital
- TS862
- Hospital Route
- Installation Boundary

Holloman
Air Force Base

Air Force Civil Engineer Center
2261 Huges Ave., Suite 163
Lackland AFB, Texas 78236-9853

Hospital Route

Jeep Target Area Skeet Range
(TS862)

FPM Remediations, Inc.

2013

NOTES:
Revision Date: 11/12/2013

Coordinate System:NAD 1983 UTM Zone 13N
Projection: Transverse Mercator
Horizontal Datum: North American 1983
False Easting: 500,000.0000
False Northing: 0.0000
Central Meridian: -105.0000
Scale Factor: 0.9996
Latitude Of Origin: 0.0000
Units: Meter

1 inch = 16,000 feet

0 2.5 5 Kilometers

0 1.5 3 Miles

N W E S

Path: C:\GIS\Projects\Holloman_AFB\Projects\Hospital_Routes\TS862_Hospital_Route.mxd

MOBILIZATION and DEMOBILIZATION
ACTIVITY HAZARD ANALYSIS

Date Prepared: 9/4/2013

Project: Holloman AFB

Job: Mobilization / Demobilization

Risk Assessment Code (RAC): L

Prepared By: David Forse

Reviewed By: Niels van Hoesel

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

Recommended Protective Clothing & Equipment:
PPE Level D: General Work Clothes, Traffic Vest, Safety Glasses, Steel Toe Boots, Hearing Protection, Work Gloves

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
Mobilize to facility and job site preparation:	Driving/Vehicle Movement (including trucks, heavy equipment)	1. Obey traffic rules· 2. 15 miles per hour is the maximum speed allowed in the work area 3. Use caution when entering roadways 4. Do not operate vehicles in unsafe conditions (e.g., on steep slopes, in deep mud) 5. Do not use cell phones when operating vehicles 6. Secure all loads, including equipment within the cab 7. Wear seat belts, including those provided in cabs of heavy equipment 8. Use caution and wear orange vests if working near active roads or around heavy equipment 9. Leave enough time to get to your destination without hurrying 10. Be aware of heavy equipment and do not park or conduct work in the blind spot of the equipment operator; remember that “blind spots” of some equipment can be very large 11. Refer to SMS 57, Vehicle Safety Program

MOBILIZATION and DEMOBILIZATION
ACTIVITY HAZARD ANALYSIS

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
All	Overhead/underground utilities	1. If overhead utilities are present in work areas, place warning signs at ground level 2. Always check for overhead utilities before using extendable equipment 3. Maintain at least one mast length or 20 feet (whichever is greater) from all power lines 4. Contact the RHSM if high voltage lines are present 5. Complete utility locates prior to intrusive work in areas where utilities have not been cleared through institutional knowledge by calling (One Call: (800) 321-ALERT and/or coordinate with site personnel) 6. Observe the area for indications of utilities
	Dust	1. Minimize generation of dust 2. Stay out of visible dust clouds 3. Wet soil if necessary to eliminate visible dust
	Noise	1. Wear hearing protection when operating or working near heavy equipment 2. Refer Hearing Conservation guidance in the HSP.
	Slips Trips and Falls	1. Make sure you have good solid footing and that walking/working surfaces are as clean and dry as possible 2. Work areas should be inspected daily and findings shall be recorded on daily inspection reports.
	Hand tools	1. Inspect tools prior to use 2. Use tools for their intended use only 3. Don't use damaged tools 4. Push, don't pull wrenches
	Biological Hazards	1. Repellents and proper clothing should be used for protection against insects including ticks, and mosquitoes. 2. Protective clothing should be used in areas where poison oak and poison ivy are present. 3. Protective clothing including long pants, and sturdy boots should be used for protections against snakes and spiders.
	Material Handling	1. Employees should use safe lifting techniques, bending at the knees and lifting with the legs. 2. Employees should use caution and not twist the back when carrying a load. 3. Mechanical devices should used to move loads when possible. 4. Protective gloves should be worn when handling materials

MOBILIZATION and DEMOBILIZATION
ACTIVITY HAZARD ANALYSIS

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
All	Cold Stress	1. Cold weather clothing and shelter should be provided as needed based on site conditions. 2. Air temperature monitoring should be done when temperatures fall below 45 degrees F.
	Heat Stress	1. Drinking water should be made available to all workers and workers should be encouraged to drink small amounts frequently. 2. Work/rest regimens shall be adjusted during hot weather.
	Extreme Weather	1. When there are warnings or indications of severe weather, conditions should be monitored and precautions taken to protect personnel.
	Fire	1. Portable fire extinguishers shall be present in all equipment and in the field trailer. 2. Fire extinguishers shall be inspected monthly. 3. Hot work permits must be obtained prior to any welding or torch cutting activities.
	Powered Machine Tools	1. Power tools shall be used, inspected and maintained according to manufacturer's recommendations. 2. Power tools designed to accommodate guards shall be equipped with such guards. 3. The electrical power control shall be provided on each power tool to make it possible for the operator to cut off the power without leaving the point of operation. 4. All electrical power tools should be connected to an in-line GFCI.

SOIL BORING/DIRECT PUSH
ACTIVITY HAZARD ANALYSIS

Date Prepared 9/4/2013

Project: Holloman AFB Job: Direct Push Soil Sampling

Risk Assessment Code (RAC): M

Prepared By: David Forse Reviewed By: Maureen Whalen

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

Recommended Protective Clothing & Equipment:
PPE Level D: General Work Clothes, Safety Glasses, Hard Hat (when overhead hazards exist), Steel Toe Boots, Chemically Resistant Gloves (when handling potentially contaminated materials)

		Probability				
		Frequent	Likely	Ocassional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
Raise mast (if utilizing drill rig/geoprobe)	Rig stability	1. Situate the rig on a flat surface 2. Use outriggers as necessary 3. Never move the rig with the mast up
	Overhead utilities	1. If overhead utilities are present in work areas, place warning signs at ground level 2. Always check for overhead utilities before raising the mast 3. Maintain at least one mast length or 20 feet (whichever is greater) from all power lines 4. Contact the PHSO if high voltage lines are present 5. Refer Utility Clearance and Isolation in HSP
Attach/detach rods and augers (hand augers included)	Lifting	1. Use the winch to lift auger flights
	Hand tools	1. Inspect tools prior to use 2. Use tools for their intended use only 3. Don't use damaged tools 4. Push, don't pull wrenches
	Pinch points	1. Never place your hand or other body parts under auger
Advance the boring (hand augers included)	Rotating equipment	1. All team members should know the location of the kill switch 2. At least two persons must be present when advancing the auger 3. Stand clear if possible 4. Do not wear loose clothing, jewelry, hair, or equipment near the auger 5. Remove cuttings with a shovel, not your hand or foot

SOIL BORING/DIRECT PUSH
ACTIVITY HAZARD ANALYSIS

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
Advance the boring (hand augers included)	Underground utilities	1. Complete utility locates prior to drilling (One Call: (800) 892-0123 and/or coordinate with site personnel) 2. Mark locations in white 3. Field verify utility locations 4. Document all utility locates 5. Observe the area for indications of utilities 6. Refer to Utility Clearance and Isolation in HSP
	Environmental Contamination (if applicable)	1. Contain cuttings in drums or plastic sheeting 2. Wear proper PPE and minimize contact with soil, sediment, groundwater, etc. 3. Work upwind of the boring 4. If unusual soil discoloration or odors are encountered, stop work, evacuate area and contact SSO; approach will need to be re-evaluated and Level C PPE may be required
	Dust (respirable silica)	1. Minimize generation of dust 2. Stay out of visible dust clouds 3. Wet soil if necessary to eliminate visible dust
	Noise	1. Wear hearing protection when operating or working near the rig 2. Refer to HSP for Hearing Conservation
Rig Maintenance	Falls	1. Fall protection is required when working at heights of greater than 6 feet (e.g., guard rails or a personal fall arrest system); refer to Fall Protection 2. Make sure you have good solid footing and that walking/working surfaces are as clean and dry as possible
	Equipment energization	1. Lockout and tagout is required if accidental energization of the rig could cause injury 2. Refer to HSP for Lockout and Tagout Safety
	Hot work	1. Clear all combustibles away from the work area 2. A fire extinguisher must be available 3. Notify the site superintendent of all hot work 4. Observe work areas for 30 minutes after hot work (“fire watch”) 5. Refer to Hot Work section of HSP.
	Chemical Hazards	1. Review material safety data sheets 2. Follow manufacturer’s instructions for use, handling and storage 3. Use recommended protective equipment 4. Label all containers
All		1. Wear proper PPE: Hardhat, safety glasses with side shields, and steel-toed boots as a minimum

SUBSURFACE SOIL SAMPLING
ACTIVITY HAZARD ANALYSIS

Date Prepared 9/4/2013

Project: Holloman AFB

Job: Surface Soil and Sediment Sampling

Risk Assessment Code (RAC): **L**

Prepared By: David Forse

Reviewed By: Maureen Whalen

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

Recommended Protective Clothing & Equipment:
PPE Level D: General Work Clothes, Safety Glasses, Steel Toe Boots, Chemical Resistant Gloves (when handling potentially contaminated materials)

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
Surface soil and sediment sampling	Ergonomics	1. Do not strain when collecting the sample 2. Use arms and shoulders, do not twist your back
	Hand tools	1. Inspect tools prior to use 2. Use tools for their intended use only 3. Don't use damaged tools
	Working near water (sediment sampling)	1. Personal flotation devices are required if working over water or adjacent to fast moving water 2. · Evaluate the potential for upstream weather events to impact dam inspection activities (e.g. increased water level and flow)
	Environmental Contamination (if applicable)	1. Contain cuttings in drums or plastic sheeting 2. Wear proper PPE and minimize contact with soil or sediment 3. Work upwind of the boring 4. If unusual soil discoloration or odors are encountered, stop work, evacuate area and contact HSM; approach will need to be re-evaluated and Level C PPE may be required

SUBSURFACE SOIL SAMPLING
ACTIVITY HAZARD ANALYSIS

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
All	Cold Stress	1. Cold weather clothing and shelter should be provided as needed based on site conditions. 2. Air temperature monitoring should be done when temperatures fall below 45 degrees F.
	Heat Stress	1. Drinking water should be made available to all workers and workers should be encouraged to drink small amounts frequently. 2. Work/rest regimens shall be adjusted during hot weather.
	Extreme Weather	1. When there are warnings or indications of severe weather, conditions should be monitored and precautions taken to protect personnel.
	Fire	1. Portable fire extinguishers shall be present in all equipment and in the field trailer. 2. Fire extinguishers shall be inspected monthly. 3. Hot work permits must be obtained prior to any welding or torch cutting activities.
	Powered Machine Tools	1. Power tools shall be used, inspected and maintained according to manufacturer's recommendations. 2. Power tools designed to accomodate guards shall be equipped with such guards. 3. The electrical power control shall be provided on each power tool to make it possible for the operator to cut off the power without leaving the point of operation. 4. All electrical power tools should be connected to an in-line GFCI.

SOIL EXCAVATION AND BACKFILL
ACTIVITY HAZARD ANALYSIS

Date Prepared 9/10/2013

Project: Holloman AFB

Job: Soil Excavation and Backfill

Risk Assessment Code (RAC): M

Prepared By: David Forse

Reviewed By: Maureen Whalen

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

Recommended Protective Clothing & Equipment:

PPE Level D
General Work Clothes, Traffic Vest, Safety Glasses, Hard Hat, Steel Toe Boots, Hearing Protection, Work Gloves as needed (EM 385-1-1 Section 05)

		Probability				
		Frequent	Likely	Ocassional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
Mobilize crew and equipment. Excavate soil Inspect spoils Backfill excavation Site restoration	Driving/Vehicle Movement (including trucks, heavy equipment)	1. Obey traffic rules· 2. 15 miles per hour is the maximum speed allowed in the work area 3. Use caution when entering roadways 4. Do not operate vehicles in unsafe conditions (e.g., on steep slopes, in deep mud) 5. Do not use cell phones when operating vehicles 6. Secure all loads, including equipment within the cab 7. Wear seat belts, including those provided in cabs of heavy equipment 8. Use caution and wear orange vests if working near active roads or around heavy equipment 9. Leave enough time to get to your destination without hurrying 10. Be aware of heavy equipment and do not park or conduct work in the blind spot of the equipment operator; remember that “blind spots” of some equipment can be very large 11. Verify back-up alarms are functional for all heavy equipment for pick-ups or SUVs with obstructed rear view, a back-up alarm or use a spotter when backing up 12. Refer to SMS 57, Vehicle Safety Program 13. Rollover protective structures (ROPS) are required on all heavy equipment with the exception of trucks used for over the road hauling.

SOIL EXCAVATION AND BACKFILL
ACTIVITY HAZARD ANALYSIS

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
All	Overhead/underground utilities	1. If overhead utilities are present in work areas, place warning signs at ground level 2. Always check for overhead utilities before using extendable equipment 3. Maintain at least one mast length or 20 feet (whichever is greater) from all power lines 4. Contact the RHSM if high voltage lines are present 5. Complete utility locates prior to intrusive work in areas where utilities have not been cleared through institutional knowledge by calling (One Call: (800) 321-ALERT and/or coordinate with site personnel) 6. Observe the area for indications of utilities
	Dust	1. Minimize generation of dust 2. Stay out of visible dust clouds 3. Wet soil if necessary to eliminate visible dust
	Noise	1. Wear hearing protection when operating or working near heavy equipment 2. Refer to SMS 26, Hearing Conservation
	Slips Trips and Falls	1. Make sure you have good solid footing and that walking/working surfaces are as clean and dry as possible 2. Work areas should be inspected daily and findings shall be recorded on daily inspection reports.
	Hand tools	1. Inspect tools prior to use 2. Use tools for their intended use only 3. Don't use damaged tools 4. Push, don't pull wrenches
	Biological Hazards	1. Repellents and proper clothing should be used for protection against insects including ticks, and mosquitoes. 2. Protective clothing should be used in areas where poison oak and poison ivy are present. 3. Protective clothing including long pants, and sturdy boots should be used for protections against snakes and spiders. 4. See SMS 047
	Material Handling	1. Employees should use safe lifting techniques, bending at the knees and lifting with the legs. 2. Employees should use caution and not twist the back when carrying a load. 3. Mechanical devices should used to move loads when possible. 4. Protective gloves should be worn when handling materials 5. See SMS 045

SOIL EXCAVATION AND BACKFILL
ACTIVITY HAZARD ANALYSIS

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
All	Heat Stress	1. Drinking water should be made available to all workers and workers should be encouraged to drink small amounts frequently. 2. Work/rest regimens shall be adjusted during hot weather. 3. See SMS 018
	Cold Stress	1. Cold weather clothing and shelter should be provided as needed based on site conditions. 2. Air temperature monitoring should be done when temperatures fall below 45 degrees F. 3. See SMS 059
	Extreme Weather	1. When there are warnings or indications of severe weather, conditions should be monitored and precautions taken to protect personnel.
	Fire	1. Portable fire extinguishers shall be present in all equipment and in the field trailer. 2. Fire extinguishers shall be inspected monthly. 3. Hot work permits must be obtained prior to any welding or torch cutting activities.
	Powered Machine Tools	1. Power tools shall be used, inspected and maintained according to manufacturer's recommendations. 2. Power tools designed to accommodate guards shall be equipped with such guards. 3. The electrical power control shall be provided on each power tool to make it possible for the operator to cut off the power without leaving the point of operation. 4. All electrical power tools should be connected to an in-line GFCI.
	Excavations	1. Excavations shall be backfilled as soon as possible after testing is complete. 2. Personnel shall not enter the excavations greater than 4 feet in depth. 3. An Excavation Competent Person shall inspect the excavations daily.
	Chemical Hazards	1. All personnel must be have current HAZWOPER training in accordance with 29 CFR 1910.120 2. MSDS shall be available for all chemicals brought to the site including fuels.

IDW MANAGEMENT
ACTIVITY HAZARD ANALYSIS

Date Prepared: 9/4/13

Project: Holloman AFB

Job: IDW Management

Risk Assessment Code (RAC): **L**

Prepared By: David Forse

Reviewed By: Maureen Whalen

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

Recommended Protective Clothing & Equipment:
PPE Level D: General Work Clothes, Safety Glasses, Steel Toe Boots, Chemical Resistant Gloves (when handling potentially contaminated materials)

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
IDW Management	Ergonomics	1. Do not strain when collecting the sample 2. Use arms and shoulders, do not twist your back
	Hand tools	1. Inspect tools prior to use 2. Use tools for their intended use only 3. Don't use damaged tools
	Environmental Contamination	1. Contain cuttings in drums or plastic sheeting 2. Wear proper PPE and minimize contact with soil 3. If unusual soil discoloration or odors are encountered, stop work, evacuate area and contact SSO; approach will need to be re-evaluated and Level C PPE may be required 4. Follow all provisions of this HSP
	Cold Stress	1. Cold weather clothing and shelter should be provided as needed based on site conditions. 2. Air temperature monitoring should be done when temperatures fall below 45 degrees F. 3. See SMS 059
	Heat Stress	1. Drinking water should be made available to all workers and workers should be encouraged to drink small amounts frequently. 2. Work/rest regimens shall be adjusted during hot weather. 3. See SMS 018
	Extreme Weather	1. When there are warnings or indications of severe weather, conditions should be monitored and precautions taken to protect personnel.
	Fire	1. Portable fire extinguishers shall be present in all equipment and in the field trailer. 2. Fire extinguishers shall be inspected monthly. 3. Hot work permits must be obtained prior to any welding or torch cutting activities.

EQUIPMENT DECONTAMINATION
ACTIVITY HAZARD ANALYSIS

Date Prepared: 9/4/13

Project: Holloman AFB

Job: Equipment Decontamination

Risk Assessment Code (RAC): **L**

Prepared By: David Forse

Reviewed By: Maureen Whalen

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

Recommended Protective Clothing & Equipment:
PPE Level D General Work Clothes, Safety Glasses, Steel Toe Boots, Hearing Protection, Work Gloves as needed

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
Equipment Decontamination	Noise	1. Wear hearing protection when operating or working near heavy equipment 2. Refer to Hearing Conservation in HSP.
	Slips Trips and Falls	1. Make sure you have good solid footing and that walking/working surfaces are as clean and dry as possible 2. Work areas should be inspected daily and findings shall be recorded on daily
	Hand tools	1. Inspect tools prior to use 2. Use tools for their intended use only 3. Don't use damaged tools 4. Push, don't pull wrenches
	Biological Hazards	1. Repellents and proper clothing should be used for protection against insects including ticks, and mosquitoes. 2. Protective clothing should be used in areas where poison oak and poison ivy are present. 3. Protective clothing including long pants, and sturdy boots should be used for protections against snakes and spiders.
	Material Handling	1. Employees should use safe lifting techniques, bending at the knees and lifting with the legs. 2. Employees should use caution and not twist the back when carrying a load. 3. Mechanical devices should used to move loads when possible. 4. Protective gloves should be worn when handling materials

EQUIPMENT DECONTAMINATION
ACTIVITY HAZARD ANALYSIS

JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS
Equipment Decontamination	Cold Stress	1. Cold weather clothing and shelter should be provided as needed based on site conditions. 2. Air temperature monitoring should be done when temperatures fall below 45 degrees F.
	Heat Stress	1. Drinking water should be made available to all workers and workers should be encouraged to drink small amounts frequently. 2. Work/rest regimens shall be adjusted during hot weather.
	Extreme Weather	1. When there are warnings or indications of severe weather, conditions should be monitored and precautions taken to protect personnel.
	Fire	1. Portable fire extinguishers shall be present in all equipment and in the field trailer. 2. Fire extinguishers shall be inspected monthly. 3. Hot work permits must be obtained prior to any welding or torch cutting activities.
	Powered Machine Tools	1. Power tools shall be used, inspected and maintained according to manufacturer's recommendations. 2. Power tools designed to accomodate guards shall be equipped with such guards. 3. The electrical power control shall be provided on each power tool to make it possible for the operator to cut off the power without leaving the point of operation. 4. All electrical power tools should be connected to an in-line GFCI.
	Chemical Hazards	1. All personnel must be have current HAZWOPER training in accordance with 29 CFR 1910.120 2. MSDS shall be available for all chemicals brought to the site including fuels.

APPENDIX E
CSE Phase II Sampling Results

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Table 5-9 PAH Sampling Results - Former Skeet Range 2 (TS859)

	Field ID:	C-LS-HL-04-SS-385[3]			C-LS-HL-04-SS-386			C-LS-HL-04-SS-387			C-LS-HL-04-SS-388			C-LS-HL-04-SB1-388			C-LS-HL-04-SB2-388		
	Sampling Date:	12/13/2011			12/13/2011			12/13/2011			12/13/2011			2/27/2012			3/1/2012		
	Lab ID:	L1120933-13			L1120933-27			L1120933-11			L1120933-05			L1203345-19			L1203620-15		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.0079	< 0.0079	U	0.008	< 0.008	U	0.0082	< 0.0082	U	0.19	< 0.19	U	0.0081	< 0.0081	U	0.0095	< 0.0095	U
Acenaphthene	0.0055	0.0079	0.0021	J	0.008	< 0.008	U	0.0082	0.0042	J	0.19	0.19		0.0081	< 0.0081	U	0.0095	< 0.0095	U
Acenaphthylene	0.0055	0.0079	< 0.0079	U	0.008	< 0.008	U	0.0082	< 0.0082	U	0.19	< 0.19	U	0.0081	< 0.0081	U	0.0095	< 0.0095	U
Anthracene	0.0022	0.0079	0.0089		0.008	< 0.008	U	0.0082	0.018		0.19	0.57		0.0081	< 0.0081	U	0.0095	0.016	
Benzo(a)anthracene	0.0167	0.0079	0.11		0.008	0.003	J	0.0082	**0.17**		0.19	**7.4**		0.0081	0.011		0.0095	0.1	
Benzo(a)pyrene	0.0165	0.0079	**0.12**		0.008	0.0048	J	0.0082	**0.18**		0.19	**7.4**		0.0081	0.012		0.0095	**0.09**	
Benzo(b)fluoranthene	0.0090	0.0079	**0.15**	J	0.008	< 0.008	U	0.0082	**0.19**	J	0.19	**8.1**	J	0.0081	0.021	J	0.0095	0.096	J
Benzo(ghi)perylene	0.0149	0.0079	0.1		0.008	0.0023	J	0.0082	0.14		0.19	5.4		0.0081	0.0086		0.0095	0.061	
Benzo(k)fluoranthene	0.0084	0.0079	0.1	J	0.008	< 0.008	U	0.0082	0.17	J	0.19	**7.4**	J	0.0081	0.0096	J	0.0095	0.096	J
Chrysene	0.0169	0.0079	0.13		0.008	0.0037	J	0.0082	0.2		0.19	8.1		0.0081	0.013		0.0095	0.11	
Dibenzo(a,h)anthracene	0.0068	0.0079	**0.029**		0.008	< 0.008	U	0.0082	**0.039**		0.19	**1.6**	J	0.0081	0.0022	J	0.0095	0.017	
Fluoranthene	0.0477	0.0079	0.18		0.008	0.0047	J	0.0082	0.28		0.19	10		0.0081	0.02		0.0095	0.2	
Fluorene	0.0055	0.0079	< 0.0079	U	0.008	< 0.008	U	0.0082	< 0.0082	U	0.19	0.056	J	0.0081	< 0.0081	U	0.0095	< 0.0095	U
Indeno(1,2,3-cd)pyrene	0.0184	0.0079	0.09		0.008	0.0052	J	0.0082	0.12		0.19	**4.8**		0.0081	0.0077	J	0.0095	0.058	
Naphthalene	0.0055	0.0079	< 0.0079	U	0.008	< 0.008	U	0.0082	0.0028	J	0.19	0.2		0.0081	< 0.0081	U	0.0095	< 0.0095	U
Phenanthrene	0.0210	0.0079	0.059		0.008	0.0023	J	0.0082	0.1		0.19	3.3		0.0081	0.0072	J	0.0095	0.09	
Pyrene	0.0322	0.0079	0.16		0.008	0.0042	J	0.0082	0.25		0.19	9.3		0.0081	0.018		0.0095	0.17	

Notes:

[1] Background sample location
[2] MS/MSD
[3] Field Duplicate Pair
[4] Field Duplicate Pair
[5] Field Duplicate Pair
[6] Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte-specific quality control criteria.
U = Undetected at the limit of detection.

Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-9 (continued) **PAH Sampling Results - Former Skeet Range 2 (TS859)**

	Field ID:	C-LS-HL-04-SS-389			C-LS-HL-04-SS-390			C-LS-HL-04-SS-391			C-LS-HL-04-SS-392			C-LS-HL-04-SS-393			C-LS-HL-04-SB1-393[2]		
	Sampling Date:	12/13/2011			12/13/2011			12/13/2011			12/13/2011			12/13/2011			2/27/2012		
	Lab ID:	L1120933-17			L1120933-08			L1120933-15			L1120933-14			L1120933-16			L1203320-30		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.0077	< 0.0077	U	0.0075	< 0.0075	U	0.042	< 0.042	U	0.0089	< 0.0089	U	1.7	< 1.7	U	0.0086	< 0.0086	U
Acenaphthene	0.0055	0.0077	< 0.0077	U	0.0075	< 0.0075	U	0.042	< 0.042	U	0.0089	< 0.0089	U	1.7	1	J	0.0086	< 0.0086	U
Acenaphthylene	0.0055	0.0077	< 0.0077	U	0.0075	< 0.0075	U	0.042	< 0.042	U	0.0089	< 0.0089	U	1.7	< 1.7	U	0.0086	< 0.0086	U
Anthracene	0.0022	0.0077	< 0.0077	U	0.0075	< 0.0075	U	0.042	< 0.042	U	0.0089	< 0.0089	U	1.7	5.5		0.0086	0.0024	J
Benzo(a)anthracene	0.0167	0.0077	0.0046	J	0.0075	0.01		0.042	**0.17**		0.0089	0.0095		1.7	**46**		0.0086	0.033	
Benzo(a)pyrene	0.0165	0.0077	0.0066	J	0.0075	0.013		0.042	**0.26**		0.0089	0.013		1.7	**42**		0.0086	**0.039**	
Benzo(b)fluoranthene	0.0090	0.0077	0.0048	J	0.0075	0.013	J	0.042	**0.34**	J	0.0089	0.012	J	1.7	**51**	J	0.0086	0.046	J
Benzo(ghi)perylene	0.0149	0.0077	0.0043	J	0.0075	0.01		0.042	0.25		0.0089	0.0099		1.7	27		0.0086	0.029	
Benzo(k)fluoranthene	0.0084	0.0077	< 0.0077	U	0.0075	0.011	J	0.042	0.24	J	0.0089	0.011	J	1.7	**37**	J	0.0086	0.035	J
Chrysene	0.0169	0.0077	0.0063	J	0.0075	0.014		0.042	0.3		0.0089	0.014		1.7	**50**		0.0086	0.041	
Dibenzo(a,h)anthracene	0.0068	0.0077	< 0.0077	U	0.0075	< 0.0075	U	0.042	**0.073**		0.0089	< 0.0089	U	1.7	**7.8**		0.0086	0.0076	J
Fluoranthene	0.0477	0.0077	0.0074	J	0.0075	0.022		0.042	0.37		0.0089	0.02		1.7	78		0.0086	0.052	
Fluorene	0.0055	0.0077	< 0.0077	U	0.0075	< 0.042	U	0.042	< 0.042	U	0.0089	< 0.0089	U	1.7	< 1.7	U	0.0086	< 0.0086	U
Indeno(1,2,3-cd)pyrene	0.0184	0.0077	0.0069	J	0.0075	0.012		0.042	**0.22**		0.0089	0.012		1.7	**25**		0.0086	0.028	
Naphthalene	0.0055	0.0077	< 0.0077	U	0.0075	< 0.0075	U	0.042	< 0.042	U	0.0089	< 0.0089	U	1.7	1.1	J	0.0086	< 0.0086	U
Phenanthrene	0.0210	0.0077	0.0029	J	0.0075	0.0077		0.042	0.088		0.0089	0.0065	J	1.7	30		0.0086	0.014	
Pyrene	0.0322	0.0077	0.0064	J	0.0075	0.019		0.042	0.33		0.0089	0.018		1.7	69		0.0086	0.048	

Notes:
[1] Background sample location
[2] MS/MSD
[3] Field Duplicate Pair
[4] Field Duplicate Pair
[5] Field Duplicate Pair
[6] Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte-specific quality control criteria.
U = Undetected at the limit of detection.
Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-9 (continued) PAH Sampling Results - Former Skeet Range 2 (TS859)

	Field ID:	C-LS-HL-04-SB2-393[4]			C-LS-HL-04-SS-394			C-LS-HL-04-SS-395			C-LS-HL-04-SS-396			C-LS-HL-04-SS-397[4]			C-LS-HL-04-SS-398[2,3]		
	Sampling Date:	3/1/2012			12/13/2011			12/13/2011			12/13/2011			12/13/2011			12/13/2011		
	Lab ID:	L1203620-09			L1120933-10			L1120933-06			L1120933-04			L1120933-24			L1120933-18		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.0091	< 0.0091	U	0.077	0.039	J	0.0089	< 0.0089	U	0.081	0.061	J	1.7	< 1.7	U	0.0078	< 0.0078	U
Acenaphthene	0.0055	0.0091	< 0.0091	U	0.077	0.12		0.0089	< 0.0089	U	0.081	0.21		1.7	1.4	J	0.0078	0.0028	J
Acenaphthylene	0.0055	0.0091	< 0.0091	U	0.077	< 0.077	U	0.0089	< 0.0089	U	0.081	< 0.081	U	1.7	< 1.7	U	0.0078	< 0.0078	U
Anthracene	0.0022	0.0091	0.0028	J	0.077	0.19		0.0089	< 0.0089	U	0.081	0.48		1.7	10		0.0078	0.0087	
Benzo(a)anthracene	0.0167	0.0091	0.023		0.077	**3.7**		0.0089	0.0036	J	0.081	**5.6**		1.7	**60**		0.0078	0.11	
Benzo(a)pyrene	0.0165	0.0091	**0.02**		0.077	**3.6**		0.0089	0.0054	J	0.081	**5.1**		1.7	**51**		0.0078	**0.13**	
Benzo(b)fluoranthene	0.0090	0.0091	0.032	J	0.077	**3.8**	J	0.0089	0.0024	J	0.081	**5.5**	J	1.7	**65**	J	0.0078	**0.15**	J
Benzo(ghi)perylene	0.0149	0.0091	0.015		0.077	2.4		0.0089	0.0027	J	0.081	3.1		1.7	34		0.0078	0.11	
Benzo(k)fluoranthene	0.0084	0.0091	0.021	J	0.077	**3.5**	J	0.0089	< 0.0089	U	0.081	**4.6**	J	1.7	**45**	J	0.0078	0.11	J
Chrysene	0.0169	0.0091	0.025		0.077	3.9		0.0089	0.0041	J	0.081	5.7		1.7	**62**		0.0078	0.13	
Dibenzo(a,h)anthracene	0.0068	0.0091	0.0036	J	0.077	**0.77**		0.0089	< 0.0089	U	0.081	**1**		1.7	**9.2**		0.0078	0.03	
Fluoranthene	0.0477	0.0091	0.049		0.077	4		0.0089	0.0069	J	0.081	7.2		1.7	110		0.0078	0.18	
Fluorene	0.0055	0.0091	< 0.0091	U	0.077	0.041	J	0.0089	< 0.0089	U	0.081	0.087		1.7	0.51	J	0.0078	< 0.0078	U
Indeno(1,2,3-cd)pyrene	0.0184	0.0091	0.014		0.077	**2.2**		0.0089	0.006	J	0.081	**3**		1.7	**30**		0.0078	0.093	
Naphthalene	0.0055	0.0091	< 0.0091	U	0.077	0.15		0.0089	< 0.0089	U	0.081	0.23		1.7	1.4	J	0.0078	0.0028	J
Phenanthrene	0.0210	0.0091	0.019		0.077	1		0.0089	0.0023	J	0.081	2.2		1.7	52		0.0078	0.053	
Pyrene	0.0322	0.0091	0.041		0.077	3.9		0.0089	0.0053	J	0.081	6.7		1.7	96		0.0078	0.16	

Notes:

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SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte-specific quality control criteria.
U = Undetected at the limit of detection.
Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-9 (continued) **PAH Sampling Results - Former Skeet Range 2 (TS859)**

	Field ID:	C-LS-HL-04-SS-842			C-LS-HL-04-SS-843			C-LS-HL-04-SS-844			C-LS-HL-04-SS-845			C-LS-HL-04-SS-846[1]			C-LS-HL-04-SS-847		
	Sampling Date:	2/27/2012			2/27/2012			2/24/2012			2/27/2012			2/27/2012			2/27/2012		
	Lab ID:	L1203345-06			L1203345-09			L1203345-12			L1203345-15			L1203320-27			L1203345-14		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.0074	< 0.0074	U	0.14	< 0.14	U	0.035	< 0.035	U	0.0074	< 0.0074	U	0.0083	< 0.0083	U	0.0069	< 0.0069	U
Acenaphthene	0.0055	0.0074	< 0.0074	U	0.14	< 0.14	U	0.035	0.017	J	0.0074	0.005	J	0.0083	< 0.0083	U	0.0069	< 0.0069	U
Acenaphthylene	0.0055	0.0074	< 0.0074	U	0.14	< 0.14	U	0.035	< 0.035	U	0.0074	< 0.0074	U	0.0083	< 0.0083	U	0.0069	< 0.0069	U
Anthracene	0.0022	0.0074	0.0052	J	0.14	< 0.14	U	0.035	0.069	J	0.0074	0.016		0.0083	< 0.0083	U	0.0069	< 0.0069	U
Benzo(a)anthracene	0.0167	0.0074	0.056		0.14	**0.44**		0.035	**0.76**		0.0074	**0.22**		0.0083	0.0034	J	0.0069	0.01	
Benzo(a)pyrene	0.0165	0.0074	**0.06**		0.14	**0.45**		0.035	**0.9**		0.0074	**0.25**		0.0083	0.0033	J	0.0069	0.011	
Benzo(b)fluoranthene	0.0090	0.0074	0.056	J	0.14	**0.47**	J	0.035	**0.95**	J	0.0074	**0.29**	J	0.0083	< 0.0083	U	0.0069	0.02	J
Benzo(ghi)perylene	0.0149	0.0074	0.047		0.14	0.34		0.035	0.72		0.0074	0.17		0.0083	0.0026	J	0.0069	0.0098	
Benzo(k)fluoranthene	0.0084	0.0074	0.053	J	0.14	0.3	J	0.035	0.71	J	0.0074	0.17	J	0.0083	< 0.0083	U	0.0069	0.0092	J
Chrysene	0.0169	0.0074	0.06		0.14	0.41		0.035	0.85		0.0074	0.27		0.0083	0.004	J	0.0069	0.013	
Dibenzo(a,h)anthracene	0.0068	0.0074	**0.016**		0.14	**0.18**		0.035	**0.17**		0.0074	**0.051**		0.0083	< 0.0083	U	0.0069	0.0023	J
Fluoranthene	0.0477	0.0074	0.1		0.14	0.66		0.035	1.2		0.0074	0.36		0.0083	0.0069	J	0.0069	0.018	
Fluorene	0.0055	0.0074	< 0.0074	U	0.14	< 0.14	U	0.035	< 0.035	U	0.0074	< 0.0074	U	0.0083	< 0.0083	U	0.0069	< 0.0069	U
Indeno(1,2,3-cd)pyrene	0.0184	0.0074	0.047		0.14	**0.36**		0.035	**0.64**		0.0074	**0.16**		0.0083	0.0026	J	0.0069	0.0083	
Naphthalene	0.0055	0.0074	< 0.0074	U	0.14	< 0.14	U	0.035	0.011	J	0.0074	0.003	J	0.0083	< 0.0083	U	0.0069	< 0.0069	U
Phenanthrene	0.0210	0.0074	0.029		0.14	0.16		0.035	0.37		0.0074	0.1		0.0083	0.0029	J	0.0069	0.0061	J
Pyrene	0.0322	0.0074	0.088		0.14	0.62		0.035	1.1		0.0074	0.34		0.0083	0.0057	J	0.0069	0.017	

Notes:
[1] Background sample location
[2] MS/MSD
[3] Field Duplicate Pair
[4] Field Duplicate Pair
[5] Field Duplicate Pair
[6] Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte-specific quality control criteria.
U = Undetected at the limit of detection.
Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-9 (continued) PAH Sampling Results - Former Skeet Range 2 (TS859)

	Field ID:	C-LS-HL-04-SS-848			C-LS-HL-04-SS-849[1,5]			C-LS-HL-04-SS-850[2,5]			C-LS-HL-04-SS-851[1]			C-LS-HL-04-SS-852[1]			C-LS-HL-04-SS-853		
	Sampling Date:	2/27/2012			2/27/2012			2/27/2012			2/27/2012			2/27/2012			2/27/2012		
	Lab ID:	L1203345-16			L1203320-28			L1203320-08			L1203320-25			L1203345-08			L1203320-21		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	< 0.0092	U	0.0098	< 0.0098	U
Acenaphthene	0.0055	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	< 0.0092	U	0.0098	< 0.0098	U
Acenaphthylene	0.0055	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	< 0.0092	U	0.0098	< 0.0098	U
Anthracene	0.0022	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	< 0.0092	U	0.0098	< 0.0098	U
Benzo(a)anthracene	0.0167	0.008	0.0022	J	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	0.0015	J	0.0092	0.017		0.0098	< 0.0098	U
Benzo(a)pyrene	0.0165	0.008	0.0022	J	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	**0.017**		0.0098	< 0.0098	U
Benzo(b)fluoranthene	0.0090	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	0.017	J	0.0098	< 0.0098	U
Benzo(ghi)perylene	0.0149	0.008	0.002	J	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	0.014		0.0098	< 0.0098	U
Benzo(k)fluoranthene	0.0084	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	< 0.0092	U	0.0098	< 0.0098	U
Chrysene	0.0169	0.008	0.003	J	0.011	< 0.011	U	0.0086	0.0033	J	0.0072	0.0023	J	0.0092	0.012		0.0098	< 0.0098	U
Dibenzo(a,h)anthracene	0.0068	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	< 0.0092	U	0.0098	< 0.0098	U
Fluoranthene	0.0477	0.008	0.004	J	0.011	0.0028	J	0.0086	0.0029	J	0.0072	0.0039	J	0.0092	0.021		0.0098	< 0.0098	U
Fluorene	0.0055	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	< 0.0092	U	0.0098	< 0.0098	U
Indeno(1,2,3-cd)pyrene	0.0184	0.008	0.002	J	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	0.017		0.0098	< 0.0098	U
Naphthalene	0.0055	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	< 0.0092	U	0.0098	< 0.0098	U
Phenanthrene	0.0210	0.008	< 0.008	U	0.011	< 0.011	U	0.0086	< 0.0086	U	0.0072	< 0.0072	U	0.0092	0.0072	J	0.0098	< 0.0098	U
Pyrene	0.0322	0.008	0.0034	J	0.011	0.0032	J	0.0086	0.0027	J	0.0072	0.0028	J	0.0092	0.018		0.0098	< 0.0098	U

Notes:

[1] Background sample location
[2] MS/MSD
[3] Field Duplicate Pair
[4] Field Duplicate Pair
[5] Field Duplicate Pair
[6] Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte-specific quality control criteria.
U = Undetected at the limit of detection.

Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-9 (continued) PAH Sampling Results - Former Skeet Range 2 (TS859)

	Field ID:	C-LS-HL-04-SS-854[6]			C-LS-HL-04-SS-855[2,6]		
	Sampling Date:	2/27/2012			2/27/2012		
	Lab ID:	L1203320-26			L1203320-22		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.01	< 0.01	U	0.0082	< 0.0082	U
Acenaphthene	0.0055	0.01	0.0055	J	0.0082	< 0.0082	U
Acenaphthylene	0.0055	0.01	< 0.01	U	0.0082	< 0.0082	U
Anthracene	0.0022	0.01	0.0034	J	0.0082	< 0.0082	U
Benzo(a)anthracene	0.0167	0.01	0.0034	J	0.0082	0.0046	J
Benzo(a)pyrene	0.0165	0.01	0.0032	J	0.0082	0.0053	J
Benzo(b)fluoranthene	0.0090	0.01	< 0.01	U	0.0082	0.015	J
Benzo(ghi)perylene	0.0149	0.01	< 0.01	U	0.0082	0.0044	J
Benzo(k)fluoranthene	0.0084	0.01	< 0.01	U	0.0082	< 0.0082	U
Chrysene	0.0169	0.01	0.0039	J	0.0082	0.006	J
Dibenzo(a,h)anthracene	0.0068	0.01	< 0.01	U	0.0082	< 0.0082	U
Fluoranthene	0.0477	0.01	0.008	J	0.0082	0.0093	
Fluorene	0.0055	0.01	0.0037	J	0.0082	< 0.0082	U
Indeno(1,2,3-cd)pyrene	0.0184	0.01	< 0.01	U	0.0082	0.0043	J
Naphthalene	0.0055	0.01	0.0075	J	0.0082	< 0.0082	U
Phenanthrene	0.0210	0.01	0.013		0.0082	0.0034	J
Pyrene	0.0322	0.01	0.0068	J	0.0082	0.0083	

Notes:
1 Background sample location
2 MS/MSD
3 Field Duplicate Pair
4 Field Duplicate Pair
5 Field Duplicate Pair
6 Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte-specific quality control criteria.
U = Undetected at the limit of detection.
Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-13 PAH Analytical Results - Jeep Target Area Skeet Range (TS862)

		Field ID:	C-LS-HL-05-SS-856			C-LS-HL-05-SB1-856			C-LS-HL-05-SB2-856			C-LS-HL-05-SS-857			C-LS-HL-05-SB1-857			C-LS-HL-05-SB2-857		
		Sampling Date:	2/27/2012			2/27/2012			2/28/2012			2/27/2012			2/27/2012			2/28/2012		
		Lab ID:	L1203320-23			L1203320-02			L1203620-01			L1203345-03			L1203320-06			L1203620-16		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**		**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055		0.074	0.042	J	0.0082	< 0.0082	U	0.0081	< 0.0081	U	0.036	< 0.036	U	0.0086	< 0.0086	U	0.0089	< 0.0089	U
Acenaphthene	0.0055		0.074	0.29		0.0082	< 0.0082	U	0.0081	< 0.0081	U	0.036	0.04		0.0086	< 0.0086	U	0.0089	< 0.0089	U
Acenaphthylene	0.0055		0.074	< 0.074	U	0.0082	< 0.0082	U	0.0081	< 0.0081	U	0.036	< 0.036	U	0.0086	< 0.0086	U	0.0089	< 0.0089	U
Anthracene	0.0022		0.074	0.98		0.0082	< 0.0082	U	0.0081	0.0018	J	0.036	0.13		0.0086	< 0.0086	U	0.0089	< 0.0089	U
Benzo(a)anthracene	0.0167		0.074	**7.2**		0.0082	0.01		0.0081	0.023		0.036	**1.4**		0.0086	< 0.0086	U	0.0089	< 0.0089	U
Benzo(a)pyrene	0.0165		0.074	**7.2**		0.0082	0.012		0.0081	**0.029**		0.036	**1.4**		0.0086	< 0.0086	U	0.0089	< 0.0089	U
Benzo(b)fluoranthene	0.0090		0.074	**6.4**	J	0.0082	0.011	J	0.0081	0.03	J	0.036	**1.4**	J	0.0086	< 0.0086	U	0.0089	< 0.0089	U
Benzo(ghi)perylene	0.0149		0.074	4.6		0.0082	0.0096		0.0081	0.02		0.036	0.91		0.0086	< 0.0086	U	0.0089	< 0.0089	U
Benzo(k)fluoranthene	0.0084		0.074	**5.4**	J	0.0082	< 0.0082	U	0.0081	0.028	J	0.036	1.2	J	0.0086	< 0.0086	U	0.0089	< 0.0089	U
Chrysene	0.0169		0.15	8.8		0.0082	0.0087		0.0081	0.035		0.036	1.7		0.0086	< 0.0086	U	0.0089	0.0032	J
Dibenzo(a,h)anthracene	0.0068		0.074	**1.4**		0.0082	0.0076	J	0.0081	0.0056	J	0.036	**0.27**		0.0086	< 0.0086	U	0.0089	< 0.0089	U
Fluoranthene	0.0477		0.15	14		0.0082	0.0095		0.0081	0.041		0.036	2.4		0.0086	< 0.0086	U	0.0089	0.0044	J
Fluorene	0.0055		0.074	0.08		0.0082	< 0.0082	U	0.0081	< 0.0081	U	0.036	< 0.036	U	0.0086	< 0.0086	U	0.0089	< 0.0089	U
Indeno(1,2,3-cd)pyrene	0.0184		0.074	**4.6**		0.0082	0.013		0.0081	0.018		0.036	**0.87**		0.0086	< 0.0086	U	0.0089	< 0.0089	U
Naphthalene	0.0055		0.074	0.055	J	0.0082	< 0.0082	U	0.0081	< 0.0081	U	0.036	0.011	J	0.0086	< 0.0086	U	0.0089	< 0.0089	U
Phenanthrene	0.0210		0.074	5.7		0.0082	0.003	J	0.0081	0.013		0.036	0.81		0.0086	< 0.0086	U	0.0089	< 0.0089	U
Pyrene	0.0322		0.15	14		0.0082	0.01		0.0081	0.048		0.036	2.5		0.0086	< 0.0086	U	0.0089	0.0037	J

Notes:

[1] MS/MSD

[2] Field Duplicate Pair

[3] Field Duplicate Pair

[4] Field Duplicate Pair

[5] Field Duplicate Pair

SS = Surface sample 0-6 inches

SB1 = Subsurface sample 6-12 inches

SB2 = Subsurface sample 12 -18 inches

J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte- specific quality control criteria.

U = Undetected at the limit of detection.

Detections < RL and > MDL reported and qualified J per DoD QSM 4.2

Detections above site background; RL is above background for some analytes

Detections above the RL and exceed RSLs

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Table 5-13 (continued) PAH Sampling Results - Jeep Target Area Skeet Range (TS862)

	Field ID:	C-LS-HL-05-SS-858			C-LS-HL-05-SB1-858			C-LS-HL-05-SB2-858			C-LS-HL-05-SS-859			C-LS-HL-05-SB1-859			C-LS-HL-05-SB2-859		
	Sampling Date:	2/27/2012			2/27/2012			2/28/2012			2/27/2012			2/27/2012			2/28/2012		
	Lab ID:	L1203320-05			L1203320-01			L1203620-05			L1203320-19			L1203320-09			L1203620-22		
Polyaromatic Hydrocarbons (mg/kg)	Site Background (mg/kg)	RL	Result	Qualifier	RL	Result	Qualifier	RL	Result	Qualifier	RL	Result	Qualifier	RL	Result	Qualifier	RL	Result	Qualifier
2-Methylnaphthalene	0.0055	0.072	< 0.072	U	0.0083	< 0.0083	U	0.0085	< 0.0085	U	0.0074	< 0.0074	U	0.0099	< 0.0099	U	0.0086	< 0.0086	U
Acenaphthene	0.0055	0.072	0.032	J	0.0083	0.0029	J	0.0085	< 0.0085	U	0.0074	< 0.0074	U	0.0099	< 0.0099	U	0.0086	< 0.0086	U
Acenaphthylene	0.0055	0.072	< 0.072	U	0.0083	< 0.0083	U	0.0085	< 0.0085	U	0.0074	< 0.0074	U	0.0099	< 0.0099	U	0.0086	< 0.0086	U
Anthracene	0.0022	0.072	0.12		0.0083	0.01		0.0085	< 0.0085	U	0.0074	0.0034	J	0.0099	< 0.0099	U	0.0086	< 0.0086	U
Benzo(a)anthracene	0.0167	0.072	**1.1**		0.0083	0.095		0.0085	< 0.0085	U	0.0074	0.033		0.0099	< 0.0099	U	0.0086	< 0.0086	U
Benzo(a)pyrene	0.0165	0.072	**1.4**		0.0083	**0.098**		0.0085	< 0.0085	U	0.0074	**0.045**		0.0099	< 0.0099	U	0.0086	< 0.0086	U
Benzo(b)fluoranthene	0.0090	0.072	**1.2**	J	0.0083	0.081	J	0.0085	< 0.0085	U	0.0074	0.048	J	0.0099	< 0.0099	U	0.0086	< 0.0086	U
Benzo(ghi)perylene	0.0149	0.072	1.1		0.0083	0.07		0.0085	< 0.0085	U	0.0074	0.035		0.0099	< 0.0099	U	0.0086	< 0.0086	U
Benzo(k)fluoranthene	0.0084	0.072	1.1	J	0.0083	0.098	J	0.0085	< 0.0085	U	0.0074	0.041	J	0.0099	< 0.0099	U	0.0086	< 0.0086	U
Chrysene	0.0169	0.072	1.6		0.0083	0.12		0.0085	< 0.0085	U	0.0074	0.054		0.0099	< 0.0099	U	0.0086	< 0.0086	U
Dibenzo(a,h)anthracene	0.0068	0.072	**0.28**		0.0083	**0.021**		0.0085	< 0.0085	U	0.0074	0.0072	J	0.0099	< 0.0099	U	0.0086	< 0.0086	U
Fluoranthene	0.0477	0.072	2.1		0.0083	0.17		0.0085	0.0029	J	0.0074	0.076		0.0099	< 0.0099	U	0.0086	0.0026	J
Fluorene	0.0055	0.072	< 0.072	U	0.0083	< 0.0083	U	0.0085	< 0.0085	U	0.0074	< 0.0074	U	0.0099	< 0.0099	U	0.0086	< 0.0086	U
Indeno(1,2,3-cd)pyrene	0.0184	0.072	**0.94**		0.0083	0.069		0.0085	< 0.0085	U	0.0074	0.034		0.0099	< 0.0099	U	0.0086	< 0.0086	U
Naphthalene	0.0055	0.072	< 0.072	U	0.0083	< 0.0083	U	0.0085	< 0.0085	U	0.0074	< 0.0074	U	0.0099	< 0.0099	U	0.0086	< 0.0086	U
Phenanthrene	0.0210	0.072	0.88		0.0083	0.063		0.0085	< 0.0085	U	0.0074	0.025		0.0099	< 0.0099	U	0.0086	< 0.0086	U
Pyrene	0.0322	0.072	2.3		0.0083	0.17		0.0085	0.0024	J	0.0074	0.074		0.0099	< 0.0099	U	0.0086	0.0028	J

Notes:
[1] MS/MSD
[2] Field Duplicate Pair
[3] Field Duplicate Pair
[4] Field Duplicate Pair
[5] Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte- specific quality control criteria.
U = Undetected at the limit of detection.
Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-13 (continued) PAH Sampling Results - Jeep Target Area Skeet Range (TS862)

	Field ID:	C-LS-HL-05-SS-890			C-LS-HL-05-SB1-890			C-LS-HL-05-SB2-890[2]			C-LS-HL-05-SS-891[1,2]			C-LS-HL-05-SS-892			C-LS-HL-05-SS-893		
	Sampling Date:	2/28/2012			2/28/2012			2/28/2012			2/28/2012			2/29/2012			2/29/2012		
	Lab ID:	L1203620-20			L1203620-23			L1203620-19			L1203620-08			L1203620-26			L1203620-03		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.36	0.61		0.043	< 0.043	U	0.03	< 0.03	U	0.39	0.65		0.24	< 0.24	U	0.01	< 0.01	U
Acenaphthene	0.0055	0.36	2.5		0.043	0.014	J	0.03	0.0088	J	0.39	2.8		0.24	0.51		0.01	< 0.01	U
Acenaphthylene	0.0055	0.36	< 0.36	U	0.043	< 0.043	U	0.03	< 0.03	U	0.39	0.09	J	0.24	< 0.24	U	0.01	< 0.01	U
Anthracene	0.0022	0.36	20		0.043	0.13		0.03	0.15		0.39	20		0.24	1.4		0.01	< 0.01	U
Benzo(a)anthracene	0.0167	3.6	**180**		0.043	**1.4**		0.03	**1**		3.9	**180**		0.24	**12**		0.01	< 0.01	U
Benzo(a)pyrene	0.0165	3.6	**160**		0.043	**1.4**		0.03	**0.88**		3.9	**170**		0.24	**12**		0.01	< 0.01	U
Benzo(b)fluoranthene	0.0090	3.6	**190**	J	0.043	**1.8**	J	0.03	**1**	J	3.9	**220**	J	0.24	**12**	J	0.01	< 0.01	U
Benzo(ghi)perylene	0.0149	3.6	110		0.043	1.1		0.03	0.6		3.9	120		0.24	6.4		0.01	< 0.01	U
Benzo(k)fluoranthene	0.0084	3.6	**130**	J	0.043	1	J	0.03	0.79	J	3.9	**130**	J	0.24	**8.8**	J	0.01	< 0.01	U
Chrysene	0.0169	3.6	**170**		0.043	1.1		0.03	0.98		3.9	**190**		0.24	14		0.01	< 0.01	U
Dibenzo(a,h)anthracene	0.0068	0.36	**35**		0.043	**0.28**		0.03	**0.16**		3.9	**33**		0.24	**2**		0.01	< 0.01	U
Fluoranthene	0.0477	3.6	330		0.043	2.4		0.03	2.1		3.9	330		0.24	20		0.01	< 0.01	U
Fluorene	0.0055	0.36	0.59		0.043	< 0.043	U	0.03	< 0.03	U	0.39	0.65		0.24	0.11	J	0.01	< 0.01	U
Indeno(1,2,3-cd)pyrene	0.0184	3.6	**100**		0.043	**0.94**		0.03	**0.56**		3.9	**110**		0.24	**6.6**		0.01	< 0.01	U
Naphthalene	0.0055	0.36	2.8		0.043	0.014	J	0.03	0.015	J	0.39	2.9		0.24	0.16	J	0.01	< 0.01	U
Phenanthrene	0.0210	3.6	140		0.043	0.88		0.03	0.95		3.9	120		0.24	7.5		0.01	< 0.01	U
Pyrene	0.0322	3.6	290		0.043	2.2		0.03	1.8		3.9	280		0.24	19		0.01	< 0.01	U

Notes:

[1] MS/MSD

[2] Field Duplicate Pair

[3] Field Duplicate Pair

[4] Field Duplicate Pair

[5] Field Duplicate Pair

SS = Surface sample 0-6 inches

SB1 = Subsurface sample 6-12 inches

SB2 = Subsurface sample 12 -18 inches

J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte- specific quality control criteria.

U = Undetected at the limit of detection.

Detections < RL and > MDL reported and qualified J per DoD QSM 4.2

Detections above site background; RL is above background for some analytes

Detections above the RL and exceed RSLs

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Table 5-13 (continued) PAH Sampling Results - Jeep Target Area Skeet Range (TS862)

	Field ID:	C-LS-HL-05-SS-894			C-LS-HL-05-SS-895			C-LS-HL-05-SS-896			C-LS-HL-05-SS-897			C-LS-HL-05-SS-898			C-LS-HL-05-SS-899		
	Sampling Date:	2/29/2012			2/29/2012			2/29/2012			2/29/2012			2/29/2012			2/29/2012		
	Lab ID:	L1203620-18			L1203620-11			L1203620-25			L1203620-04			L1203620-13			L1203620-12		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.01	< 0.01	U	0.0099	< 0.0099	U	0.043	< 0.043	U	0.0077	< 0.0077	U	0.0072	< 0.0072	U	0.0087	< 0.0087	U
Acenaphthene	0.0055	0.01	< 0.01	U	0.0099	< 0.0099	U	0.043	0.031	J	0.0077	< 0.0077	U	0.0072	< 0.0072	U	0.0087	< 0.0087	U
Acenaphthylene	0.0055	0.01	< 0.01	U	0.0099	< 0.0099	U	0.043	< 0.043	U	0.0077	< 0.0077	U	0.0072	< 0.0072	U	0.0087	< 0.0087	U
Anthracene	0.0022	0.01	0.0047	J	0.0099	< 0.0099	U	0.043	0.18		0.0077	< 0.0077	U	0.0072	< 0.0072	U	0.0087	< 0.0087	U
Benzo(a)anthracene	0.0167	0.01	0.068		0.0099	0.0062	J	0.043	**1.3**		0.0077	0.004	J	0.0072	0.0046	J	0.0087	< 0.0087	U
Benzo(a)pyrene	0.0165	0.01	**0.097**		0.0099	0.0076	J	0.043	**1.3**		0.0077	0.004	J	0.0072	0.005	J	0.0087	< 0.0087	U
Benzo(b)fluoranthene	0.0090	0.01	0.1	J	0.0099	0.018	J	0.043	**1.3**	J	0.0077	< 0.0077	U	0.0072	< 0.0072	U	0.0087	< 0.0087	U
Benzo(ghi)perylene	0.0149	0.01	0.07		0.0099	0.0063	J	0.043	0.69		0.0077	0.0032	J	0.0072	0.0053	J	0.0087	< 0.0087	U
Benzo(k)fluoranthene	0.0084	0.01	0.094	J	0.0099	< 0.0099	U	0.043	1.1	J	0.0077	< 0.0077	U	0.0072	< 0.0072	U	0.0087	< 0.0087	U
Chrysene	0.0169	0.01	0.087		0.0099	0.0095	J	0.043	1.7		0.0077	0.0053	J	0.0072	0.006	J	0.0087	< 0.0087	U
Dibenzo(a,h)anthracene	0.0068	0.01	**0.017**		0.0099	< 0.0099	U	0.043	**0.22**		0.0077	< 0.0077	U	0.0072	< 0.0072	U	0.0087	< 0.0087	U
Fluoranthene	0.0477	0.01	0.16		0.0099	0.012		0.043	2.5		0.0077	0.0079		0.0072	0.008		0.0087	< 0.0087	U
Fluorene	0.0055	0.01	< 0.01	U	0.0099	< 0.0099	U	0.043	< 0.043	U	0.0077	< 0.0077	U	0.0072	< 0.0072	U	0.0087	< 0.0087	U
Indeno(1,2,3-cd)pyrene	0.0184	0.01	0.065		0.0099	0.0054	J	0.043	**0.71**		0.0077	0.0029	J	0.0072	0.0035	J	0.0087	< 0.0087	U
Naphthalene	0.0055	0.01	< 0.01	U	0.0099	< 0.0099	U	0.043	0.014	J	0.0077	< 0.0077	U	0.0072	< 0.0072	U	0.0087	< 0.0087	U
Phenanthrene	0.0210	0.01	0.047		0.0099	0.0041	J	0.043	1.2		0.0077	0.0032	J	0.0072	0.0033	J	0.0087	< 0.0087	U
Pyrene	0.0322	0.01	0.16		0.0099	0.011		0.043	2.5		0.0077	0.0069	J	0.0072	0.0072		0.0087	< 0.0087	U

Notes:

[1] MS/MSD
[2] Field Duplicate Pair
[3] Field Duplicate Pair
[4] Field Duplicate Pair
[5] Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte- specific quality control criteria.
U = Undetected at the limit of detection.

Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-13 (continued) PAH Sampling Results - Jeep Target Area Skeet Range (TS862)

	Field ID:	C-LS-HL-05-SS-900			C-LS-HL-05-SS-901			C-LS-HL-05-SS-902			C-LS-HL-05-SS-903			C-LS-HL-05-SS-904			C-LS-HL-05-SS-905		
	Sampling Date:	2/29/2012			2/29/2012			3/2/2012			3/2/2012			3/2/2012			3/2/2012		
	Lab ID:	L1203620-07			L1203620-21			L1203792-16			L1203792-21			L1203792-20			L1203792-18		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.072	< 0.072	U	0.0083	< 0.0083	U	0.094	< 0.094	U	0.0076	< 0.0076	U	0.28	< 0.28	U	0.014	< 0.014	U
Acenaphthene	0.0055	0.072	< 0.072	U	0.0083	< 0.0083	U	0.094	0.027	J	0.0076	< 0.0076	U	0.28	< 0.28	U	0.014	< 0.014	U
Acenaphthylene	0.0055	0.072	< 0.072	U	0.0083	< 0.0083	U	0.094	< 0.094	U	0.0076	< 0.0076	U	0.28	< 0.28	U	0.014	< 0.014	U
Anthracene	0.0022	0.072	< 0.072	U	0.0083	< 0.0083	U	0.094	0.18		0.0076	< 0.0076	U	0.28	< 0.28	U	0.014	0.013	J
Benzo(a)anthracene	0.0167	0.072	< 0.072	U	0.0083	0.0051	J	0.094	**1**		0.0076	0.0029	J	0.28	< 0.28	U	0.014	0.13	
Benzo(a)pyrene	0.0165	0.072	< 0.072	U	0.0083	0.0055	J	0.094	**0.83**		0.0076	0.0029	J	0.28	< 0.28	U	0.014	**0.14**	
Benzo(b)fluoranthene	0.0090	0.072	< 0.072	U	0.0083	0.014	J	0.094	**0.9**	J	0.0076	0.011	J	0.28	< 0.28	U	0.014	**0.18**	J
Benzo(ghi)perylene	0.0149	0.072	< 0.072	U	0.0083	0.0041	J	0.094	0.48		0.0076	0.0025	J	0.28	< 0.28	U	0.014	0.12	
Benzo(k)fluoranthene	0.0084	0.072	< 0.072	U	0.0083	< 0.0083	U	0.094	0.79	J	0.0076	< 0.0076	U	0.28	< 0.28	U	0.014	0.1	J
Chrysene	0.0169	0.072	< 0.072	U	0.0083	0.0067	J	0.094	1.1		0.0076	0.0035	J	0.28	< 0.28	U	0.014	0.17	
Dibenzo(a,h)anthracene	0.0068	0.072	< 0.072	U	0.0083	< 0.0083	U	0.094	**0.14**		0.0076	< 0.0076	U	0.28	< 0.28	U	0.014	**0.029**	
Fluoranthene	0.0477	0.072	< 0.072	U	0.0083	0.0096		0.094	2.2		0.0076	0.0043	J	0.28	< 0.28	U	0.014	0.24	
Fluorene	0.0055	0.072	< 0.072	U	0.0083	< 0.0083	U	0.094	< 0.094	U	0.0076	< 0.0076	U	0.28	< 0.28	U	0.014	< 0.014	U
Indeno(1,2,3-cd)pyrene	0.0184	0.072	< 0.072	U	0.0083	0.0035	J	0.094	**0.48**		0.0076	0.0023	J	0.28	< 0.28	U	0.014	0.1	
Naphthalene	0.0055	0.072	< 0.072	U	0.0083	< 0.0083	U	0.094	0.028	J	0.0076	< 0.0076	U	0.28	< 0.28	U	0.014	< 0.014	U
Phenanthrene	0.0210	0.072	< 0.072	U	0.0083	0.0036	J	0.094	0.99		0.0076	0.0019	J	0.28	< 0.28	U	0.014	0.093	
Pyrene	0.0322	0.072	< 0.072	U	0.0083	0.0096		0.094	1.8		0.0076	0.0038	J	0.28	< 0.28	U	0.014	0.22	

Notes:
[1] MS/MSD
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[3] Field Duplicate Pair
[4] Field Duplicate Pair
[5] Field Duplicate Pair
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SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte- specific quality control criteria.
U = Undetected at the limit of detection.
Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-13 (continued) PAH Sampling Results - Jeep Target Area Skeet Range (TS862)

	Field ID:	C-LS-HL-05-SS-906			C-LS-HL-05-SS-907			C-LS-HL-05-SS-919[3]			C-LS-HL-05-SS-920[1,3]			C-LS-HL-05-SS-921			C-LS-HL-05-SS-922		
	Sampling Date:	3/2/2012			3/2/2012			3/2/2012			3/2/2012			3/2/2012			3/2/2012		
	Lab ID:	L1203792-17			L1203792-23			L1203792-03			L1203792-02			L1203792-04			L1203792-13		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.015	< 0.015	U	0.0074	< 0.0074	U	0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Acenaphthene	0.0055	0.015	< 0.015	U	0.0074	< 0.0074	U	0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Acenaphthylene	0.0055	0.015	< 0.015	U	0.0074	< 0.0074	U	0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Anthracene	0.0022	0.015	< 0.015	U	0.0074	< 0.0074	U	0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Benzo(a)anthracene	0.0167	0.015	0.039		0.0074	0.013		0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Benzo(a)pyrene	0.0165	0.015	**0.052**		0.0074	0.014		0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Benzo(b)fluoranthene	0.0090	0.015	0.071	J	0.0074	0.021	J	0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Benzo(ghi)perylene	0.0149	0.015	0.052		0.0074	0.013		0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Benzo(k)fluoranthene	0.0084	0.015	0.055	J	0.0074	0.014	J	0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Chrysene	0.0169	0.015	0.062		0.0074	0.016		0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Dibenzo(a,h)anthracene	0.0068	0.015	0.011	J	0.0074	0.003	J	0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Fluoranthene	0.0477	0.015	0.08		0.0074	0.026		0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	0.0023	J	0.0082	< 0.0082	U
Fluorene	0.0055	0.015	< 0.015	U	0.0074	< 0.0074	U	0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Indeno(1,2,3-cd)pyrene	0.0184	0.015	0.044		0.0074	0.011		0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Naphthalene	0.0055	0.015	< 0.015	U	0.0074	< 0.0074	U	0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Phenanthrene	0.0210	0.015	0.021		0.0074	0.0085		0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	< 0.0082	U	0.0082	< 0.0082	U
Pyrene	0.0322	0.015	0.078		0.0074	0.023		0.0084	< 0.0084	U	0.0083	< 0.0083	U	0.0082	0.0021	J	0.0082	< 0.0082	U

Notes:

[1] MS/MSD
[2] Field Duplicate Pair
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[4] Field Duplicate Pair
[5] Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte- specific quality control criteria.
U = Undetected at the limit of detection.
Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-13 (continued) PAH Sampling Results - Jeep Target Area Skeet Range (TS862)

	Field ID:	C-LS-HL-05-SS-923			C-LS-HL-05-SS-924[4]			C-LS-HL-05-SS-925[1,4]			C-LS-HL-05-SS-926			C-LS-HL-05-SS-927			C-LS-HL-05-SS-928[5]		
	Sampling Date:	3/2/2012			3/2/2012			3/2/2012			3/2/2012			3/2/2012			3/2/2012		
	Lab ID:	L1203792-12			L1203792-11			L1203792-05			L1203792-06			L1203792-07			L1203792-08		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.008	< 0.008	U	0.0079	< 0.0079	U	0.0079	< 0.0079	U	0.009	< 0.009	U	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Acenaphthene	0.0055	0.008	0.004	J	0.0079	0.0018	J	0.0079	0.0028	J	0.009	< 0.009	U	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Acenaphthylene	0.0055	0.008	< 0.008	U	0.0079	< 0.0079	U	0.0079	< 0.0079	U	0.009	< 0.009	U	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Anthracene	0.0022	0.008	0.015		0.0079	0.0099		0.0079	0.01		0.009	0.0021	J	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Benzo(a)anthracene	0.0167	0.008	**0.18**		0.0079	0.12		0.0079	**0.15**		0.009	0.011		0.0081	< 0.0081	U	0.0081	< 0.0081	U
Benzo(a)pyrene	0.0165	0.008	**0.22**		0.0079	**0.16**		0.0079	**0.2**		0.009	0.0098		0.0081	< 0.0081	U	0.0081	< 0.0081	U
Benzo(b)fluoranthene	0.0090	0.008	**0.2**	J	0.0079	**0.19**	J	0.0079	**0.2**	J	0.009	0.019	J	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Benzo(ghi)perylene	0.0149	0.008	0.16		0.0079	0.14		0.0079	0.16		0.009	0.0086	J	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Benzo(k)fluoranthene	0.0084	0.008	0.2	J	0.0079	0.15	J	0.0079	0.17	J	0.009	0.0095	J	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Chrysene	0.0169	0.008	0.25		0.0079	0.21		0.0079	0.24		0.009	0.013		0.0081	< 0.0081	U	0.0081	< 0.0081	U
Dibenzo(a,h)anthracene	0.0068	0.008	**0.041**		0.0079	**0.034**		0.0079	**0.039**		0.009	< 0.009	U	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Fluoranthene	0.0477	0.008	0.32		0.0079	0.3		0.0079	0.31		0.009	0.022		0.0081	0.0028	J	0.0081	< 0.0081	U
Fluorene	0.0055	0.008	< 0.008	U	0.0079	< 0.0079	U	0.0079	< 0.0079	U	0.009	< 0.009	U	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Indeno(1,2,3-cd)pyrene	0.0184	0.008	**0.15**		0.0079	0.13		0.0079	**0.15**		0.009	0.0069	J	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Naphthalene	0.0055	0.008	< 0.008	U	0.0079	< 0.0079	U	0.0079	< 0.0079	U	0.009	< 0.009	U	0.0081	< 0.0081	U	0.0081	< 0.0081	U
Phenanthrene	0.0210	0.008	0.12		0.0079	0.095		0.0079	0.097		0.009	0.011		0.0081	< 0.0081	U	0.0081	< 0.0081	U
Pyrene	0.0322	0.008	0.34		0.0079	0.28		0.0079	0.31		0.009	0.019		0.0081	0.0024	J	0.0081	< 0.0081	U

Notes:

[1] MS/MSD
[2] Field Duplicate Pair
[3] Field Duplicate Pair
[4] Field Duplicate Pair
[5] Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
J = Estimated: The analyte was positively identified; the quantitation is an estimation and / or the quantitation is an estimation due to discrepancies in meeting certain analyte- specific quality control criteria.
U = Undetected at the limit of detection.
Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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Table 5-13 (continued) PAH Sampling Results - Jeep Target Area Skeet Range (TS862)

	Field ID:	C-LS-HL-05-SS-929[1,5]			C-LS-HL-05-SS-930			C-LS-HL-05-SS-931		
	Sampling Date:	3/2/2012			3/2/2012			3/2/2012		
	Lab ID:	L1203792-10			L1203792-15			L1203792-09		
Polyaromatic Hydrocarbons (mg/kg)	**Site Background (mg/kg)**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**	**RL**	**Result**	**Qualifier**
2-Methylnaphthalene	0.0055	0.0082	< 0.0082	U	0.008	< 0.008	U	0.0081	< 0.0081	U
Acenaphthene	0.0055	0.0082	< 0.0082	U	0.008	< 0.008	U	0.0081	< 0.0081	U
Acenaphthylene	0.0055	0.0082	< 0.0082	U	0.008	< 0.008	U	0.0081	< 0.0081	U
Anthracene	0.0022	0.0082	< 0.0082	U	0.008	< 0.008	U	0.0081	< 0.0081	U
Benzo(a)anthracene	0.0167	0.0082	< 0.0082	U	0.008	0.0037	J	0.0081	< 0.0081	U
Benzo(a)pyrene	0.0165	0.0082	< 0.0082	U	0.008	0.0041	J	0.0081	< 0.0081	U
Benzo(b)fluoranthene	0.0090	0.0082	< 0.0082	U	0.008	< 0.008	U	0.0081	< 0.0081	U
Benzo(ghi)perylene	0.0149	0.0082	< 0.0082	U	0.008	0.0035	J	0.0081	< 0.0081	U
Benzo(k)fluoranthene	0.0084	0.0082	< 0.0082	U	0.008	< 0.008	U	0.0081	< 0.0081	U
Chrysene	0.0169	0.0082	< 0.0082	U	0.008	0.0051	J	0.0081	< 0.0081	U
Dibenzo(a,h)anthracene	0.0068	0.0082	< 0.0082	U	0.008	< 0.008	U	0.0081	< 0.0081	U
Fluoranthene	0.0477	0.0082	< 0.0082	U	0.008	0.0065	J	0.0081	< 0.0081	U
Fluorene	0.0055	0.0082	< 0.0082	U	0.008	< 0.008	U	0.0081	< 0.0081	U
Indeno(1,2,3-cd)pyrene	0.0184	0.0082	< 0.0082	U	0.008	0.0033	J	0.0081	< 0.0081	U
Naphthalene	0.0055	0.0082	< 0.0082	U	0.008	< 0.008	U	0.0081	< 0.0081	U
Phenanthrene	0.0210	0.0082	< 0.0082	U	0.008	0.0023	J	0.0081	< 0.0081	U
Pyrene	0.0322	0.0082	< 0.0082	U	0.008	0.0062	J	0.0081	< 0.0081	U

Notes:

[1] MS/MSD
[2] Field Duplicate Pair
[3] Field Duplicate Pair
[4] Field Duplicate Pair
[5] Field Duplicate Pair
SS = Surface sample 0-6 inches
SB1 = Subsurface sample 6-12 inches
SB2 = Subsurface sample 12 -18 inches
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U = Undetected at the limit of detection.
Detections < RL and > MDL reported and qualified J per DoD QSM 4.2
Detections above site background; RL is above background for some analytes
Detections above the RL and exceed RSLs

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