Department of Energy
National Nuclear Security Administration
Sandia Field Office
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Albuquerque, NM 87185

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Hazardous Waste Bureau

Mr. Dave Cobrain
Manager
Permits Management Program
Hazardous Waste Bureau
New Mexico Environment Department
2905 Rodeo Park Dr. East, Bldg. 1
Santa Fe, NM 87505

Subject: Submittal of Updated Reference Documents Cited in the Mixed Waste Landfill Long-Term Monitoring and Maintenance Plan for Sandia National Laboratories/New Mexico, Environmental Protection Agency Identification Number NM5890110518

Dear Mr. Cobrain:

The Department of Energy/National Nuclear Security Administration and Sandia Corporation (Sandia) are submitting the enclosed updated reference documents to the New Mexico Environment Department. This submittal is required within 30 days of the document effective date, which occurred on June 16, 2014.

This submittal includes two documents used by Sandia personnel to validate analytical data from contract laboratories and conduct activities related to sampling Mixed Waste Landfill soil-gas wells, respectively. The third document is the health and safety plan for groundwater monitoring. The updated reference documents are:

AOP 00-03 *Data Validation Procedure for Chemical and Radioactive Data*
FOP 08-22 *Soil Vapor Sampling*
PLA 05-09 *Groundwater Monitoring Health and Safety Plan*

If you have questions, please contact John Weckerle of my staff at (505) 845-6026.

Sincerely,

James W. Todd
Assistant Manager for Engineering

Enclosure
cc: See Page 2

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14-538- 583529

Submittal of Updated Reference Documents Cited in the Mixed Waste Landfill Long-Term Monitoring and Maintenance Plan

Sandia National Laboratories
Albuquerque, New Mexico

CERTIFICATION STATEMENT

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 ensure 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 or imprisonment for knowing violations.

Michael W. Hazen, Vice-President
Sandia Corporation
Albuquerque, New Mexico
Operator

26 Jun 2014
Date signed

James W. Todd, Assistant Manager
U.S. Department of Energy
National Nuclear Security Administration
Sandia Field Office
Owner

08 July 2014
Date signed

Enclosure A

Updated Reference Documents Cited in the Mixed Waste Landfill Long-Term Monitoring and Maintenance Plan for Sandia National Laboratories/New Mexico

June 2014

AOP 00-03 Data Validation Procedure for Chemical and Radiochemical Data
FOP 08-22 Soil Vapor Monitoring
PLA 05-09 Groundwater Monitoring Health and Safety Plan

SANDIA NATIONAL LABORATORIES
LONG TERM STEWARDSHIP DEPARTMENT (4142)

DATA VALIDATION PROCEDURE FOR CHEMICAL AND RADIOCHEMICAL DATA for the SAMPLE MANAGEMENT OFFICE

AOP 00-03
Revision 4

Prepared By: Monica Dymerski — **Date:** 05/09/14
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Mike Moore
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Approved: Pam Puissant — **Date:** 6/16/14
Pam Puissant,
Manager, Long Term Stewardship, 4142

Author: *How frequently does this document need to be reviewed and/or revised?*	*Every 3 years*
Manager: *Does this document need to be tracked?*	*Yes*

EFFECTIVE DATE: _____

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List of Appendices

Revision History

Revision	Effective Date	Summary of Changes
0	12/21/1999	New document
1	12/08/2003	Update to reflect current administrative changes
2	7/16/2007	Update to reflect current administrative changes
3	5/16/2011	EPA Method updates and references. Process changes for the automatic upload of the data validation qualifiers and the necessary QC steps associated with the new data processing procedure.
4	6/16/2014	Update to responsibilities of SMO personnel, and EPA methods and references. Clarified data validation requirements associated with mass spectra acceptability and TIC evaluation. Added language that states an MS/MSD is not required for an isotope dilution analysis. Included additional method-specific analytical requirements for Organic LC/MS/MS (PPCP) and Inorganic Anions by Ion Chromatography.

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ACRONYMS AND ABBREVIATIONS

SNL acronyms that do not need to be defined in SNL Memos

AA	Atomic Absorption
ARCOC	Analysis Request and Chain of Custody
BOD	Biological Oxygen Demand
CAS	Chemical Abstract Service
CB	Chlorinated Biphenyl
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CF	Calibration Factor
CLP	Contract Laboratory Program
CRA	RLV for AA Methods
CRDL	RLV for Cyanide Methods
CRI	RLV for ICP-AES, ICP-MS, and LC/MS/MS Methods
CVR	Contract Verification Review
DL	Detection Limit
DDD	Dichlorodiphenyldichloroethane
DDE	Dichlorodiphenyldichloroethylene
DDT	Dichlorodiphenyltrichloroethane
DOE	U.S. Department of Energy
DRO	Diesel Range Organics
EB	Equipment Blank
EDD	Electronic Data Deliverable
EICP	Extracted Ion Current Profile
EPA	U.S. Environmental Protection Agency
FB	Field Blank
FWHM	Full-width Half-maximum
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
GPC	Gel Permeation Chromatography
GRO	Gasoline Range Organics
HE	High Explosive
HPLC	High-Performance Liquid Chromatography
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry

ACRONYMS AND ABBREVIATIONS (continued)

IC	Ion Chromatography
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
ICS	Interference Check Standard
ICS A	Interference Check Sample Solution A
ICS AB	Interference Check Sample Solution AB
ICV	Initial Calibration Verification
IS	Internal Standard
LC/MS/MS	Liquid Chromatography/Mass Spectrometry/Mass Spectrometry
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOC	Level of Chlorination
LRMS	Low Resolution Mass Spectrometry
MB	Method Blank
MDA	Minimum Detectable Activity
MDC	Minimum Detectable Concentration
MDL	Method Detection Limit
MIR	Manual Integration Review
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDC	Mass Spectrometry Data Centre
NBS	National Bureau of Standards
NIH	National Institute of Health
NIST	National Institute of Standards and Technology
NNSA	National Nuclear Security Administration
OP	Operating Procedure
OPR	Ongoing Precision and Recovery
PAH	Polyaromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PCDD	Polychlorinated Dibenzodioxin
PCDF	Polychlorinated Dibenzofuran
PPCP	Pharmaceuticals and Personal Care Products
PQL	Practical Quantitation Limit
PS	Post-digestion spike

ACRONYMS AND ABBREVIATIONS (concluded)

QC	Quality Control
RER	Replicate Error Ratio
RF	Response Factor
RL	Reporting Limit
RLV	Reporting Limit Verification
RPD	Relative Percent Difference
RRF	Relative Response Factor
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RT	Retention Time
SD	Serial Dilution
SDG	Sample Delivery Group
SMO	Sample Management Office
SNL/NM	Sandia National Laboratories/New Mexico
SOW	Statement of Work
SVOC	Semivolatile Organic Compound
SW	Solid Waste (EPA procedure number)
TAL	Target Analyte List
TB	Trip Blank
TCDF	Tetrachlorodibenzofuran
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TPH	Total Petroleum Hydrocarbon
TPU	Total Propagated Uncertainty
VOC	Volatile Organic Compound

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MEASUREMENTS AND SYMBOLS

°C	degrees Centigrade
>	greater than
≥	greater than or equal to
<	less than
≤	less than or equal to
%	percent
%D	percent difference
%R	percent recovery
%RSD	percent relative standard deviation
±	plus or minus
X	times
σ	sigma
amu	atomic mass units
r	correlation coefficient
r^2	coefficient of determination
kev	kilo electron volt
kg	kilogram
L	liter
m/e	mass/charge ratio
ug	microgram
mg	milligram
mL	milliliter(s)
ppb	parts per billion
ppbv	parts per billion by volume

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1.0 PURPOSE

The purpose of data validation is to identify, through the evaluation of supporting documentation, those data that do not meet the expected precision and accuracy of an analytical method. This procedure presents the guidelines used to evaluate chemical (organic and inorganic) and/or radiochemical analytical data acquired in support of environmental and waste management activities. The purpose of the procedure is to consistently qualify data using defined criteria; however, it is not intended to eliminate the need for professional judgment in evaluating the data quality. The data validator may be more or less stringent in evaluating the results based on experience and familiarity with the analytical techniques, historical data, sample matrices, or intended use of the data. The product of this procedure is a data validation report that includes information regarding the overall quality of the data and the resulting data qualifiers. When variations in the application of data qualifiers are warranted, the justification and rationale will be explained in the data validation report.

2.0 SCOPE AND OWNERSHIP

2.1 Scope

This procedure specifically covers the validation of chemical or radiochemical analytical results from environmental methods required for Sandia National Laboratories/New Mexico (SNL/NM) Sample Management Office (SMO) decisions but may be used by other organizations as appropriate. The format is based on analytical techniques, standard reporting protocols used by the laboratories, and the general format followed by the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) functional guidelines. Additions and modifications were made to address analyses requested by the SNL/NM SMO customers. Any apparent redundancies between sections, is stylistically intentional for the sake of completeness and accuracy. Qualification of data performed under this procedure does not replace any data usability review for specific project use.

2.2 Ownership

The SNL/NM SMO owns this operating procedure (OP). The SMO is responsible for maintaining and revising this OP as necessary. Any comments or suggestions for improvement should be forwarded to the SMO.

3.0 RESPONSIBLE INDIVIDUALS AND ORGANIZATIONS

This section describes the responsibilities of SNL/NM personnel and contractors regarding this OP.

3.1 Department Manager

The department manager is responsible for providing programmatic guidance leading to the development of this OP and the following:

- Reviewing and approving the OP.

- Acting as liaison to the U.S. Department of Energy (DOE) and the National Nuclear Security Administration (NNSA)/Sandia Field Office on data validation issues.
- Ensuring that resources are available to perform tasks in compliance with this OP.

3.2 SMO Technical Lead

The SMO technical lead is responsible for the operations and activities conducted within the SMO. The principal responsibilities of the SMO technical lead include but are not limited to the following:

- Updating this OP.
- Managing the validation contract, acting as the Sandia Delegated Representative, reviewing routine performance assessments, and conducting general contract oversight.
- Providing oversight of the data review and validation process.
- Ensuring this OP is implemented for review and validation of analytical data provided by the contract laboratories when data validation is requested.
- Developing and maintaining processes that ensure the necessary documentation, to perform data review and validation, is made available to the laboratory oversight/data validation contractor.

3.3 SMO QA Coordinator

The SMO QA Coordinator is responsible for:

- Providing project data quality assurance guidance.
- Ensuring that this procedure is distributed to the appropriate personnel for project/program use.
- Ensuring that sufficient quality checks are in place to maintain the integrity of the SMO sample information management and analytical result database.
- Documenting non-conformances and corrective actions in accordance with the applicable SMO-QAPP.
- Interfacing with the Records Management Coordinator for maintenance of project documentation and to resolve record management concerns for storage and maintenance of sampling and analysis records.

3.4 SMO Project Coordinator

The SMO project coordinator is responsible for coordinating efforts associated with SMO analytical services. The principal responsibilities of the SMO project coordinator include but are not limited to the following:

- Acting as a point of contact between Task/Project Leaders, the analytical laboratories, and the laboratory oversight/data validation contractor.
- Performing the Contract Verification Review (CVR) and completing form SMO 2012-CVR on analytical data packages from the contract laboratories pursuant to "Procedure for Completing the Contract Verification Review," (SMO-05-03) and the SNL/NM Contract Statement of Work (SOW) for Analytical Laboratories (contract SOW).
- Transmitting and tracking electronically the complete analytical data package along with the CVR form to the laboratory oversight/data validation contractor.

- Implementing and follow-up of all nonconformances and corrective actions with the contract analytical laboratories and laboratory oversight/data validation contractor.
- Processing the EDD that includes the data validation qualifiers, into the Environmental Data Management System (EDMS), pursuant to "Procedure for Electronic Data Deliverable (EDD) Processing," (SMO-05-04).
- Performing quality control (QC) checks on all data validation results by reviewing the report and comparing the results to the data validation qualifiers captured on the EDD.
- Transmitting the complete analytical data package to SMO project/data management staff for final archiving.

3.5 SMO Project/Data Management Staff

The SMO project/data management staff is responsible for:

- Ensuring compliance with the "SMO Data Management Plan," (AOP 95-44).
- Receiving and processing analytical data packages
- Managing data flow and data storage, including both hardcopy paper records from field activities and analytical laboratories, and electronic data relating to sample tracking or analytical results.
- Forwarding the complete and final analytical data package and electronic data to the SNL/NM Records Center for archiving.

3.6 Laboratory Oversight/Data Validation Contractor

The Laboratory Oversight/Data Validation Contractor is responsible for:

- Performing data validation in accordance with this OP.
- Requesting data corrections or additional information needed from the contract analytical laboratories and notifying SMO of the request.
- Notifying SMO of all data determined as rejected ("R" coded) according to this OP.
- Communicating non-compliance issues to the SMO technical lead and/or SMO project coordinator(s) and ensuring that nonconformances (e.g., incorrect or missing analytical information) are adequately addressed.
- Completing the data validation report including checklist(s) and if applicable generating validation EDD files (see Section 5.1).
- Communicating with the SMO customer or designated representative when data review and validation is complete and returning the complete data package to the SMO.
- Verifying implementation of laboratory corrective action plans.
- Performing laboratory oversight as directed by the SMO.

4.0 PROCEDURE

Data are evaluated using common quality parameters from QC measurements specified in the methods and the SNL/NM contract SOW. These parameters are compared to statistically derived or regulatory method criteria to estimate the quality of the results. The quality parameters are measures of the analytical precision and accuracy, potential contamination both from the field and from the laboratory, sample matrix effects, and sample inhomogeneity. The laboratory may define the acceptance criteria as long as they meet or exceed those specifically defined within the method or contract. The appropriateness of acceptance criteria generated by the laboratory should be evaluated periodically by the SMO.

Qualification is based on minimal reporting requirements and does not address method or contract compliance requirements, except within the context of QC data. Complete method and contract compliance cannot generally be performed using only the laboratory data package and should be done during on-site assessments at the laboratory where all supporting documentation is available.

If any QC element for a method is not provided, the data validation report must document that the QC data are missing and any qualification is at the discretion of the data validator. A QC failure for an analyte that results in "R" coded (unusable) data due to matrix problems (e.g., matrix interference that cannot be alleviated by acceptable clean-up procedures) brings the appropriateness of the analytical method into question. As a result, the data validation report must document that analysis by another acceptable method or modification of the existing method may be necessary.

4.1 General

This section provides the portions of the method for reviewing QC data that are pertinent to both chemical (organic and inorganic) and radiochemical analytical data.

4.1.1 CVR

The SMO is responsible for conducting a CVR of analytical data packages delivered from the contract laboratories using the SMO "Procedure for Completing the Contract Verification Review," (SMO-05-03).

Criteria: A CVR form shall be included with the analytical data package that specifically addresses the Analysis Request and Chain of Custody (ARCOC), receipt of samples by the laboratory, and the technical, QC, and reporting requirements imposed upon the analytical laboratory through the contract SOW.

Evaluation	Action
The CVR form should be checked to confirm: ARCOC (SMO 2012-ARCOC) and laboratory login information have been reviewed,	Report any discrepancies and/or anomalies associated with the CVR form to SMO.

Evaluation (concluded)	Action (concluded)
missing samples and sample container irregularities are discussed, preservation and hold time deficiencies are indicated, appropriate target analyte lists (TALs) and contract-required laboratory qualifiers are used, results are reported, in correct units, for all analytes requested, all radiochemistry results include the calculated total propagated uncertainty (TPU), the required detection limits (DL) are reported and clearly defined or an explanation of why they were not met is given, all outstanding reporting issues are resolved, any request for an amended report from the laboratory has been received, and signatures and dates are present indicating CVR was completed.	

4.1.2 QC Exemptions

Various filter materials may be submitted for analysis. Matrix spike (MS) and replicate sample analysis requirements shall not apply to filter materials because representative splits of these samples are generally not obtainable. All other QC criteria shall apply to the analysis of filters.

The requirements for reanalysis for QC failures are waived when insufficient sample remains. A detailed discussion of that condition shall be included in the laboratory case narrative when it is encountered.

Acidity, alkalinity, biological oxygen demand (BOD), color, corrosivity, dissolved oxygen, gravimetric oil and grease, hardness, ignitability, pH, titrimetric sulfide, conductivity, all of the solids methods, and turbidity analyses are generally exempt from the general inorganic QC requirements.

Criteria: The analyses referenced directly above shall be controlled according to the method QC and/or the laboratory's QC policies. In general, one or more of the following should be included:

- Blank; result less than (<) the method detection limit (MDL).
- Laboratory control sample (LCS); measured value within plus or minus (±) 20 percent (%) of known value.
- Duplicate; relative percent difference (RPD) <25%.
- Independent calibration check standard; result within ± 10% of true value.

Note: Blanks (method blank [MB]/field blank [FB])/equipment blank [EB]) are not applicable for acidity by titration, alkalinity, conductivity, flash point, pH, and specific gravity. In the Blanks section of the data validation report, document that the blank result was reported but not assessed for data validation.

Evaluation	**Action**
If there are any QC failures for any of the analyses listed above,	qualify sample results associated with QC failures according to the appropriate requirements in Section 4.6, Procedure for Inorganic Data Validation. **Note**: Sample results shall not be qualified due to the lack of QC data. QC exemptions shall be discussed in the data validation report.

4.1.3 Holding Times

Samples must be extracted and analyzed within EPA-specified holding times for results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding time criteria must be considered to be suspect. Holding times must be evaluated to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis.

Solid materials, such as soils, that are being analyzed for radioisotopes or metals are generally exempt from qualification for exceeded holding times. The reviewer should evaluate the stability of the analyte and half-life, if applicable, and qualify based on professional judgment.

In the case of organic analyses, regulatory holding times are set by analytical method and do not address the stability of individual compounds; however, studies have been conducted to determine the stability of many of the commonly requested volatile compounds in preserved water. In some special cases, water samples for the analysis of volatile organic compounds (VOCs) are from sampling events that cannot be resampled, and rejecting non-detects may be very detrimental to the program. In these

special cases, the holding time qualification guidelines given in Appendix A may be used, but the data validation report must clearly state that the evaluation and qualification were not performed using regulatory holding time guidelines.

Criteria: All samples will be extracted and analyzed within specified holding times, per Appendix B.

Evaluation	Action
If a holding time infraction is <5% of the holding time criteria,	sample results may be accepted without qualification based on professional judgment. **Note:** Consideration should be given to the relevant holding time requirement; for example, "days" versus "hours."
If holding times are exceeded and preservation requirements are not met (see Section 4.1.4),	qualify all associated detects as "J" and all associated non-detects as "R."
If samples were analyzed after their holding time had expired but within 2 times (X) the specified holding time,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If samples were analyzed beyond 2X the specified holding time,	qualify all associated detects as "J" and all associated non-detects as "R."
If samples were analyzed within holding time and reanalyzed out of holding time due to a QC failure and…	
the original and reanalysis calibration, sample, and QC data are provided and the sample results are similar,	accept the results of the reanalysis without holding time qualification.
the original or reanalysis calibration, sample, and QC data are not provided, or the sample results of the original analysis and the reanalysis are not similar,	qualify all associated detects as "J" and all associated non-detects as "UJ."

4.1.4 Preservation (chemical and temperature)

Samples must be preserved according to EPA-specified criteria for results to be considered reflective of total concentrations. Analytical data generated outside of the specified preservation criteria must be considered to be suspect. The data validation report shall include a discussion of any preservation violations and a discussion supporting any qualifications.

Many organic compounds and most metals and radioisotopes are not affected by temperature variations up to ambient temperature and are generally not qualified. VOCs and mercury are subject to analyte loss at elevated temperatures.

Criteria: All samples shall be preserved and shipped under conditions specified in Appendix B.

Samples for metals or radiochemical analysis that were received without the required chemical preservation but that were preserved by the laboratory after receipt generally do not require qualification if the samples were allowed to equilibrate at least 16 hours before a sample aliquot is taken.

Evaluation	**Action**
If samples were received outside the temperature criteria,	all associated detects may be qualified as "J" and all associated non-detects may be qualified as "UJ" or "R" using professional judgment (see below).
If temperature violations occur for VOCs and/or mercury,	qualify all associated detects as "J" and all associated non-detects as "UJ." Non-detects for VOCs may be qualified as "R" if extreme temperature violations occur.
If samples preserved by the laboratory upon receipt were not allowed to equilibrate after laboratory preservation or if no documentation shows the samples were allowed to equilibrate,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If samples were received without the required preservation and were not preserved by the laboratory after receipt,	qualify all associated detects as "J" and all associated non-detects as "R."

4.1.5 Calibration Points

Generally, it is not acceptable to remove points from the calibration curve unless the points are at the high or low ends of the curve. For the purpose of meeting calibration criteria, if a point is removed from the low end, the practical quantitation limit (PQL) must be adjusted accordingly. If a point is removed from the high end, the linear calibration range must be adjusted accordingly. Whenever a point is removed, it must be clearly documented in the instrument log. All initial calibration (ICAL) points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

The laboratory may remove ICAL data points that are not the low or high points of the average or linear/quadratic curve, if the reason can be clearly documented. Acceptable reasons include

misinjection of the standard or minor instrument failure for the particular data point. Notify the laboratory project manager if no such documentation is present.

4.1.6 Calibration for QC Samples

If any QC samples are analyzed using a different ICAL than that of the field samples, the laboratory must include a calibration report from the calibration affecting the QC samples. This calibration data shall only be used to evaluate the QC samples and only if the QC samples fail to meet recovery or RPD acceptance criteria. The laboratory is not required to report calibration data associated with QC samples from another sample delivery group (SDG).

4.1.7 Blank Hierarchy

The general hierarchy for application of qualifiers due to blank contamination is 1) instrument blank, 2) preparation blank or MB, and 3) FB, EB, or trip blank (TB). As a general guideline, if the instrument blank is contaminated, then associated detected results in field samples, MB, FBs, EBs, and TBs that are analyzed in the same analytical run may be qualified. If the preparation blank is contaminated, all associated detected results in samples prepared with that blank may be qualified even if the samples are analyzed in different runs. If an FB or EB is contaminated, all associated detected results in samples collected during the same sampling event may be qualified. If a TB is contaminated, all associated detected results in samples transported in the same container (cooler) may be qualified. Professional judgment must be employed to determine the effect of multiple blank contaminations upon the quality of field sample data.

4.1.8 Blank Normalization

Because sample aliquot values (masses or volumes) seldom vary significantly within a batch, the laboratory generally assigns a representative aliquot value to the MB. When a sample has a significantly different aliquot size than that of the MB, a detected MB result needs to be normalized to the detected sample result before a comparison can be performed for blank assessment. The blank data are normalized to the sample results using the following equation:

Normalized blank concentration = (blank concentration) X (blank aliquot value/sample aliquot value)

It should be noted that the blank analyses might not involve the same weights, volumes, and/or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5X and 10X criteria, such that the total amount of contamination is actually compared.

4.1.9 Field Duplicates

Field duplicate samples may be collected and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates which measure only laboratory performance. It is expected that solid or waste duplicate results will have a greater variance than water matrices due to inhomogeneity.

If samples are identified as field duplicates, document the occurrence in the data validation report and state that there are no "required" review criteria for field duplicate analyses comparability.

4.1.10 Sample-Specific External Standard Recovery

In lieu of an internal standard (IS) addition, an addition of a known quantity of material to a second sample aliquot may be used to calculate sample results. To evaluate external standard recovery (standard addition), the spike amount and spike recovery must be reported.

Criteria: Recovery guidelines for external standard recovery shall be 50% to 105%. The quantity of external standard used should be adequate to provide a reasonable confidence level in the measured recovery; that is, the spike level should be greater than (>) the indigenous level.

Note: For samples that require dilution the evaluation uses the concentration of the diluted result not the corrected result.

Evaluation	**Action**
If the measured sample result is >2X the external standard spike added,	qualify all associated results as "J."
If the measured sample result is >4X the external standard spike added,	qualify all associated results as "R."
If the recovery is >105% but less than or equal to (≤) 125%,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
If the recovery is >125%,	qualify all associated non-detects as "R." Associated detects may be qualified "J-" or "R" based on professional judgment.
If the recovery is <50% but greater than or equal to (≥) 20%,	qualify all associated detects as "J+."
If the recovery is <20%,	qualify all associated non-detects as "R."

4.1.11 Rounding Rules and Significant Figures

If the figure is ≥5, round up; otherwise, round down. For example, 11.443 is rounded down to 11.44, and 11.455 is rounded up to 11.46. If a series of multiple operations is to be performed (i.e., add, subtract, divide, and/or multiply), all figures are carried through the calculations. The final answer is rounded to the proper number of significant figures. Before evaluating a number for being in control or out of control of a certain limit, the number evaluated shall be rounded using these rounding rules to the significance reported for that limit. For example, if the acceptance limit is ±10% of the true value, then a calculated percent recovery (%R) of 110.46 shall be reported as 110%, which is within the acceptance limits of 90% to 110%. On the other hand, a calculated %R of 110.50 shall be reported as 111%, which is not within the 90% to 110% acceptance limits.

Blank qualifications with an associated numerical value should be recorded with no more than three significant figures for values ≥100, and no more than two significant figures for values <100 in the data validation report; for example, 125U, 18U, 9.9U, 0.32U or 0.032U.

4.1.12 Special Laboratory Flags

"X" Flags

Criteria: The laboratory or analyst may have reason to believe that the result for a specific analysis has a high probability of being a false positive due to interferences. In this case, the laboratory shall qualify the result as "X" and narrate the justification for the flag. Generally, use of the "X" flag is restricted to use in conjunction with additional data such as spectral matching or results from another analytical technique. The raw data and case narrative should be reviewed to determine if they agree with the identification of a false positive.

Evaluation	Action
When evaluating the "X" qualifier, if it is determined that the interference is the most significant source of the instrument response (i.e., if the detect is primarily a false positive or if it is a detect with a very high bias),	qualify detects determined to be primarily false positives as "R" and detects determined to have very high bias as "NJ+." Include a thorough discussion supporting the qualification in the data validation report.

4.1.13 Analytical Methods

The laboratory shall follow the requirements specified in the analytical methods and those specified in the contract SOW. When these requirements are not met, reanalysis is required. In those cases where reanalysis cannot occur, the failure to reanalyze will be discussed in the case narrative. This discussion should also be included in the data validation report. See Appendix C for data reporting requirements.

4.1.14 Calculations

Criteria: Laboratories will generally use commercial software whenever possible. Spreadsheets and laboratory developed software are required to be verified and uniquely identified, and shall include a revision number (i.e., be under version control). Reverification of commercial software and other software is not routinely required. Hand-calculated data or data calculated from a spreadsheet or other software not under version control must be verified by the random recalculation of some of the results. Hand calculated results and spreadsheets

should have all required formulas and data included in the package. In addition, any spreadsheet that is not under version control should be brought to the attention of the SMO.

Evaluation	Action
If results cannot be regenerated using the reported data,	require a formal corrective action by the laboratory.
If results are verified by recalculation using reported data,	discuss the recalculation in the data validation report.

Criteria: Laboratories are required to calculate the RPD between the MS and matrix spike duplicate (MSD) using the actual results (Solid Waste [SW]-846 Method 8000C). CLP and some other programs use calculation routines, which calculate the RPD using the %Rs.

RPD = (MS %R - MSD %R)/[(MS %R + MSD %R)/2]

This does not give an equivalent result as that obtained using the SW-846 formula (see Section 6.3 below) when the sample contains indigenous analyte. When the RPD is calculated using %Rs, the results will need to be recalculated before the evaluation is performed.

Evaluation	Action
If results are recalculated using the correct data,	discuss the recalculation in the data validation report.

4.1.15 Reanalysis

The laboratory may perform a reanalysis on one or more samples because of QC failures. This may occur because of MS failures, or it may occur because a small subset such as the acid fraction in semivolatile organic compound (SVOC) analysis had QC failures for the first analysis and the second analysis was performed outside the method-specific holding time. Based on professional judgment the laboratory is to report only the best data set on the certificate of analysis (COA). All supporting documentation concerning a reanalysis will be provided in the miscellaneous data section of the analytical data package.

4.1.16 Manual Integration

Manual integration review (MIR) is typically outside the scope of routine validation. When MIR is required by the program, it must be performed in accordance with standard operating procedures.

Manual integration is used to correct improper integration performed by the instrument software, not for the purpose of meeting QC criteria. While MIR is not normally required for data validation,

manually integrated peaks may be reviewed based on professional judgment or whenever QC problems indicate it may be necessary

Evaluation	Action
If a manual integration was not documented correctly or was performed incorrectly or does not meet one or more of the criteria given,	request confirmation from the laboratory of the need for regeneration of data. Data may be qualified as "J" or "R" based on professional judgment.

4.1.17 Failed Batch QC

Occasionally the batch QC sample (i.e., MS, LCS, blank, etc.) will fail and the individual QC sample will also fail sample-specific QC parameters (i.e., ISs, surrogates, etc.) The usefulness of the QC data from these batch QC samples is based on professional judgment for minor excursions. However, significant failures where the QC sample fails both sample parameters (i.e., surrogates, etc.) and batch parameters (e.g., analyte) require that the batch QC data be rejected and the batch be treated as if it did not include the batch QC sample. That is, the samples are qualified as if no QC sample was run with the batch.

4.1.18 MS/MSD, LCS/LCSD and Replicates

Occasionally the laboratory may analyze for replicates, matrix spike/matrix spike duplicate (MS/MSD) pairs, and/or LCS/laboratory control sample duplicate (LCSD) pairs, presenting more than one measure of precision. If the sample has little or no indigenous analyte, the MS/MSD RPD is the best indicator of precision. If the sample has significant indigenous analyte, the replicate is the best indicator of precision. As a general rule, the replicate precision is used if the indigenous analyte is >2X or 3X the MS spike concentration. The LCS/LCSD RPD should only be used as a measure of precision in the absence of both MS/MSD and replicate analyses.

More than one measurement of precision is not assessed for the same sample/analyte. The data validation report should include a discussion on which measure of precision was used for assessment and why.

4.1.19 MS/MSD

Occasionally the laboratory may dilute before spiking or may run the MS/MSD pairs at a reduced volume. For example, the sample aliquot will be 1000 milliliters (mL) while the MS aliquot is 500 ml. In this case, if the extract volume is the same for both the sample aliquot and the MS aliquot, the RPD is still a good measure of precision, but the %R is not a good measure of accuracy and matrix effect. At a minimum, this issue should be noted in the data validation report. If the final volume of the MS aliquot was adjusted for sample size (in this case, adjusted to half the sample extract volume), it should be noted that the laboratory may have adjusted the extract volume to account for a smaller sample aliquot. In this case, the MS %R is a good measure of accuracy and matrix effect.

MS samples that require dilution due to matrix should not be used to evaluate associated sample data unless the relative dilution factor between the MS and the field samples is ≤5, in which case there is still significant sample matrix similarity between the MS and field samples. If the MS sample is not used to evaluate sample data, the sample results should be qualified for lack of accuracy and/or precision data, as applicable, if specified by the program.

Evaluation	Action
If the MS/MSD relative dilution factor is >5 compared to the samples,	qualify all associated detects as "J" and all associated non-detects as "UJ."

4.1.20 MS/MSD with Elevated Analyte Concentration Requirements

When the sample used for the MS/MSD has an analyte concentration >4X the analyte spike concentration and the MS and/or MSD %R is out of limits, sample results should be qualified due to a lack of matrix-specific accuracy data. Matrix-specific precision can still be assessed using the MS/MSD RPD. If a post-digestion spike (PS) is also performed, it can be used to assess matrix-specific accuracy data for the analytes not evaluated using the MS/MSD. The 4X rule also applies to the PS; however, its analyte spike concentration may be higher than that of the MS/MSD. The PS recovery limits are usually narrower than the MS recovery limits. The MS and MSD results may be used in conjunction with other QC results to determine the need for qualification of the data.

Evaluation	Action
If the sample used for MS/MSD has an analyte concentration >4X the analyte spike concentration and the MS and/or MSD %R for that analyte is out of limits,	qualify all associated detects as "J" and all associated non-detects as "UJ."

4.1.21 Blank Qualification with QC Failures

Data may be qualified as a non-detect (U) based on blank contamination and have other QC failures. While the general approach is to qualify the sample result as a non-detect with no further qualification, other quality issues should be considered to determine if additional qualification is warranted. For example, if the LCS had very low recovery, the actual sample result may be below the blank result because of poor recovery, not just because of blank contamination. In this case, the result may be qualified "UJ" rather than "U." In general, samples with results that are qualified "U" or "UJ" due to blank contamination are not rejected. Justification for additional qualification must be explained in the data validation report.

4.1.22 Initial Dilutions

Initial dilutions may be required due to high indigenous analyte concentrations. For multi-analyte determinations where initial dilutions are required to keep from saturating the detector, the DLs and

reporting limits (RL) must be adjusted for the initial dilution. In addition, the matrix effect of the over-range analyte on the other analytes being measured cannot be determined.

Evaluation	Action
If all target analytes are reported from the same diluted run,	all associated detects may be qualified as "J" and all associated non-detects may be qualified as "UJ" based on professional judgment.

4.1.23 Reporting Limit Verification (RLV)

Data from independent RLV standards may be used to additionally evaluate the intercept. Acceptable RLVs may be used to minimize qualification based on professional judgment. An acceptable curve with a low standard at the RL does not meet this requirement. The RLV must be the measurement of an independent standard.

4.1.24 Filtered Samples

Water samples may be submitted as both field filtered and unfiltered aliquots. When it is evident that both a filtered and an unfiltered sample are submitted, both results will be reviewed. The analyte concentrations for the filtered portion should be ≤ the unfiltered portion.

Evaluation	Action
If the analyte concentrations of the filtered portion are generally > that of the unfiltered portion,	contact the laboratory to determine if a sample mix-up has occurred.
If the analyte concentrations of the filtered portion are generally > that of the unfiltered portion and the reason cannot be identified,	document the problem and contact the technical data support for further direction.

4.1.25 Sample Contamination

There may be instances where little or no contamination is present in the associated blank, but qualification of the sample due to contamination is deemed necessary. Contamination introduced in a diluent is one example. Although it is not always possible to determine, evidence of this occurrence can be identified when contaminants are found in the diluted sample result but are absent in the undiluted sample.

Evaluation	Action
If it is determined that the sample contamination is from a source not identified in the blank,	qualify the results for that analyte as "R" and discuss such circumstances in the data validation report.

4.2 Procedure for Gas Chromatography/Mass Spectrometry (GC/MS) Validation

The requirements addressed within this section are applicable to all GC/MS analytical techniques.

4.2.1 Instrument Tuning for GC/MS

Tuning and performance criteria are established to ensure mass resolution, identification; and, to some degree, sensitivity. These criteria are not sample-specific. Conformance is determined using standard materials. Therefore, these criteria should be met in all circumstances.

Criteria: The GC/MS tune shall be evaluated daily. The relative abundance criteria listed in the appropriate method must be met.

Evaluation	Action
If tunes are not run daily or if all abundance criteria are not met,	contact the laboratory for immediate corrective action and use professional judgment to determine which data should be used. The following actions are suggested: qualify all associated detects as “J” and all associated non-detects as “UJ.”
If multiple QC failures also occurred,	qualify all results as “R.”

4.2.2 Calibration

Initial Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the TAL. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run and of producing a linear curve. In the absence of, or in addition to, method-specific calibration acceptance criteria, the following general calibration acceptance criteria should be applied.

The laboratory may establish a calibration curve using either the linear regression (linear curve) approach or the average response factor (RF) approach. If both approaches are used to quantify and report target analytes within the same data package, calibration is to be assessed on an analyte-by-analyte basis.

Criteria: GC/MS instrument calibration shall be performed using a minimum of five calibration standards unless otherwise specified by the method. If calibration curves are used, five standards are required for a linear (first-order) calibration model, six standards are required for a quadratic (second-order) model, and seven standards are required for a third-order polynomial. Higher order curves (second order and higher) should not normally be used. If the laboratory uses a higher-order equation to establish a calibration curve, it should be evaluated for appropriate application.

Evaluation	Action
If an insufficient number of calibration standards was used,	qualify all associated detects as "J" and all associated non-detects as "UJ."

RFs

Criteria: RFs are a measure of the slope of the calibration relationship and assumes that the curve passes through the origin. Under ideal conditions, the factors will not vary with the concentration of the standard that is injected into the instrument. In practice, some variation is to be expected.

When the variation, measured as the percent relative standard deviation (%RSD), is ≤15%, the use of the linear model is appropriate and the calibration curve can be assumed to be linear and to pass through the origin. This criterion is derived from SW-846 GC/MS Methods 8260B/8260C and 8270C/8270D.

As a general rule, the amount of IS should produce an instrument response (e.g., area counts) that is no more than 100X that produced by the lowest concentration of the least responsive target compound associated with the IS. This should result in a minimum RF of no <0.01 for the least responsive target compound.

The %RSD for the RFs obtained from the five ICAL standards must be ≤15% and the average RF shall be ≥ the method-specified minimum RF for each compound. The minimum RFs for the system performance check compounds per method SW-846 8260B/8260C (VOC) are:

- Bromoform 0.10
- Chlorobenzene 0.30
- Chloromethane 0.10
- 1,1-Dichloroethane 0.10
- 1,1,2,2-Tetrachloroethane 0.30

Compounds (VOC and SVOC) without specified minimum RFs will be >0.05.

Evaluation	Action
If the average RF for any target compound is < the specified minimum RF, or <0.05 if no minimum is specified,	qualify all associated detects as "J" and all associated non-detects as "UJ" if the average RF is ≥0.01 and as "R" if the average RF is <0.01.
If the %RSD for any target compound is...	
>15% but ≤40%,	qualify all associated detects as "J" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>40% but ≤60%,	qualify all associated detects as "J" and all associated non-detects as "UJ."
>60%,	qualify all associated detects as "J" and all associated non-detects as "R."

Linear Curves

Criteria: The coefficient of determination (r^2) of the ICAL curve shall be ≥0.99 and have a slope ≥ the method-specified minimum RF for each compound. Compounds without method-specified minimum RFs shall have a slope ≥0.05. The absolute value of the intercept shall be ≤3X the MDL.

Note: The sample results may be reported with non-detects at the MDL or at the PQL value. See below for appropriate evaluation.

Note: The intercept reported in the instrument calibration report may not be in appropriate units. When the intercept is not in appropriate units, the instrument conversion routine may be needed to evaluate the intercept.

For calibrations using most commercial data system software the intercept in concentration units is:

Concentration Intercept = (b)(CIS)

Where:

b = reported intercept

CIS = concentration of IS (on-column conc. on quant. report)

Evaluation	**Action**
If the slope for any target compound is < the minimum RF, or <0.05 if no minimum is specified,	qualify all associated detects as "J" and all associated non-detects as "UJ" if the slope is ≥0.01 and as "R" if the slope is <0.01.
If the r^2 for any target compound is... <0.99 but ≥0.90, <0.90 but ≥0.80, <0.80,	 qualify all associated detects as "J" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ." qualify all associated detects as "J" and all associated non-detects as "UJ." qualify all associated detects as "J" and all associated non-detects as "R."
If the intercept for any target compound is positive and > the MDL,	qualify all associated detects <3X the intercept as "J+."
When results are reported at the MDL: If the intercept for any target compound is negative with an absolute value… > the MDL but ≤3X the MDL, >3X the MDL,	 qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ." qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "R."
When results are reported at the PQL: If the intercept for any target compound is negative with an absolute value... > the MDL but ≤2X the PQL, >2X the PQL,	 qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ." qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "R."

4.2.3 Calibration Verification

Compliance requirements for satisfactory initial and continuing instrument calibration verification are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the TAL. Initial calibration verification (ICV) independently verifies the calibration and continuing calibration verification (CCV) establishes the 12-hour relative RFs on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

Criteria: An ICV standard must be analyzed immediately following an ICAL.

The ICV standard analysis results are not required to be reported in the data package unless the samples in the SDG were analyzed after the ICAL standard but before a CCV standard analysis was performed. In this case, the ICV percent difference (%D) is assessed according to the calibration verification criteria described below for the associated samples. If a CCV is analyzed prior to samples and ICV data are also reported in the package, both the ICV %D and the appropriate CCV %D are to be assessed as described below. If both ICV %D and CCV %D infractions occur, the worst infraction should be evaluated for result qualification. A CCV standard must be analyzed:

(1) if analysis continues for longer than 12 hours, and

(2) at the beginning of each additional 12-hour period.

The laboratory is allowed to perform corrective action and reanalyze the CCV once after a failure. If more than two CCVs were analyzed to obtain a passing CCV, then the calibration was not verified and the calibration verification frequency criteria were not met.

Evaluation	Action
If the ICV and CCV standards were not analyzed at the proper frequency, or if either a required ICV or CCV was not analyzed, or if all target compounds were not present in any ICV or CCV standard,	qualify all associated detects as “J” and all associated non-detects as “UJ.”
If all required ICVs and CCVs were not analyzed,	qualify all associated detects as “J” and all associated non-detects as “R.”

RFs

Criteria: The %D between the ICV and/or CCV RFs and the average RFs obtained from the ICAL shall be calculated according to the formula in Section 6.3 and must be ≤20%.

Evaluation	Action
If the %D was reported with the wrong sign (e.g., + %D for a negative bias),	document the occurrence in the data validation report and assess any infractions using the correct sign.
If the %D between an ICAL RF and an ICV or CCV RF for any target compound is...	
>20% and positive (high bias),	qualify all associated detects as "J+."
>20% but ≤40% and negative (low bias),	qualify all associated detects as "J-" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>40% but ≤60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
>60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "R."

Linear Curves

Criteria: The %D (see Section 6.3) between the ICV and/or CCV standard concentrations and their true values must be ≤ 20%.

Evaluation	Action
If the %D was reported with the wrong sign (e.g., +%D for negative bias),	document the occurrence in the data validation report and assess any infractions using the correct sign.
If the %D between the measured ICV and/or CCV concentrations and their true values for any target compound is...	
>20% and positive (high bias),	qualify all associated detects as "J+."
>20% but ≤40% and negative (low bias),	qualify all associated detects as "J-" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ.

Evaluation (concluded)	Action (concluded)
>40% but ≤60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
>60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "R."

4.2.4 Blanks

The purpose of laboratory (or field) blank analysis is to determine the essence and magnitude of contamination resulting from laboratory (or field) activities.

The criteria for evaluation of blanks apply to any blank associated with the samples and include MBs, and, if submitted, EBs, FBs, and TBs. Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. For purposes of evaluating multiple blanks, each preparation batch may be considered an independent event in evaluating MBs, and each sampling event may be considered an independent event for evaluating EBs, FBs, and TBs.

The result for any compound detected in the sample (other than those listed below), that was also detected in any associated blank, must be qualified when the sample concentration is <5X the blank concentration. For the following compounds, the results are qualified when the sample concentration is <10X the blank concentration.

Common laboratory contaminants:

- Methylene chloride
- Acetone
- Toluene
- 2-butanone
- Common phthalate esters (e.g., bis(2-ethylhexyl)phthalate, di-n-octyl phthalate)

Criteria: The concentration of each target analyte found in the blank must be < the associated MDL. The sample results must not be corrected by subtracting any blank value. If QC problems with any blank exist, all data associated with the case must be carefully evaluated to determine whether there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

Evaluation	Action
If a compound detected in a blank is also detected in a field sample,	qualify the sample result for that compound in accordance with the scenarios given below.
If gross contamination (e.g.., saturated peaks by GC/MS) exists,	qualify results for all affected compounds as "R" due to interference.
If inordinate numbers of target compounds are found at low levels in the blank(s),	Discuss the presence of these compounds in the data validation report as it may be indicative of a problem at the laboratory. **Note:** Similar consideration should be given to tentatively identified compounds (TICs) that are found in both the sample and its associated blank(s) (see Section 4.2.13).

The following are examples of application of the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Scenario 1

The sample result is > the PQL but is <5X or 10X the blank result.

Qualification

Rule	10X	5X
Blank Result	7	7
PQL	5	5
Sample Result	30	30
Qualified Sample Result	30U	30U

In the example for the 10X rule, qualify sample results <70 (or 10 X 7) as a non-detect ("U") at the reported value. In the case of the 5X rule, qualify sample results <35 (or 5 X 7) as a non-detect ("U") at the reported value.

Scenario 2

The sample result is < the PQL, and is also <5X or 10X the blank result.

Qualification

Rule	10X	5X
Blank Result	6	6
PQL	5	5
Sample Result	4J	4J
Qualified Sample Result	5U	5U

In the example for the 10X rule, qualify sample results <60 (or 10 X 6) as a non-detect ("U") at the PQL. In the case of the 5X rule, qualify sample results <30 (or 5 X 6) as a non-detect ("U") at the PQL.

Note: Data are not reported as 4U, as this would be reported as a DL below the PQL.

The PQL may not be reported and it may not be possible to determine the PQL from the data. In these cases, qualify the contaminated sample result as "U" at 5X (or 10X) the blank concentration.

Note: In some instances, the laboratory may adjust their MDLs to account for low-level common laboratory contaminants. In these cases, it may be possible to have a low level detection in a blank that would be considered a non-detect when compared to the adjusted MDL, resulting in the blank data being reported as a non-detect (PQL U). This may result in sample results that are above the MDL but <5X or 10X the actual blank concentration not being qualified. In instances where it is believed that there is low-level contamination of common laboratory contaminants that are not identified in the blank, the sample results may be qualified as "NJ" based on professional judgment and discussed in the data validation report.

Scenario 3

The sample result is > the PQL, and is also >5X or 10X the blank result.

Qualification

Rule	10X	5X
Blank Result	10	10
PQL	5	5
Sample Result	120	120
Unqualified Sample Result	120	120

For both the 10X and 5X rules, the sample result exceeded the adjusted blank result of 100 (or 10 X 10) and 50 (or 5 X 10), respectively. Therefore, this sample result is not qualified.

4.2.5 Surrogate Recovery

Laboratory performance for individual samples is evaluated by means of surrogate spikes. All samples are spiked with surrogate compounds prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interference and high concentrations of analytes. Because the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific surrogate results is frequently subjective and demands analytical experience and professional judgment. In addition, surrogate recoveries can be influenced by the success in recoveries of the ISs. The evaluation of surrogate recoveries and ISs should be done concurrently. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

Criteria: Sample and blank surrogate recoveries must be within limits specified by the laboratory. Surrogate compound recoveries shall be calculated using the procedure described in SW-846 Method 8000C. Reported recoveries shall be accompanied by the applicable acceptance limits. No qualification with respect to surrogate recovery is placed on data unless one or more of the following occurs:

1) at least two surrogates are out of specification in the base/neutral fraction or acid fraction (SVOC analysis),

2) one surrogate is out of specification in the volatile fraction (VOC analysis), or

3) any surrogate has < 10 %R.

Under these three conditions, there should be a reanalysis.

Note: The common acid fraction analytes (SVOC) are all phenols; all cresols; benzoic acid; dichlorophenoxyacetic acid; dinoseb; and hexachlorophene.

Note: When there are unacceptable surrogate recoveries followed by successful reanalysis, the laboratories are required to report only the successful run.

See Appendix D for general guidelines for surrogate recovery limits.

Note: Results from spiked or replicate QC samples that have surrogate recoveries < 10% cannot be used to evaluate associated sample results. Sample results should be qualified for lack of accuracy and/or precision data, as applicable, if specified by the program.

Evaluation	Action
If surrogate recovery acceptance criteria were not reported in the data package,	request amended data from the laboratory.

Evaluation (continued)	**Action (continued)**
If, based on professional judgment, the laboratory's internal acceptance criteria are excessively wide or biased,	notify the program manager.
If an initial dilution was performed on any sample and at least one surrogate recovery is < the lower acceptance limit but ≥10%, or all surrogate recoveries are <10% and the results for one or more compounds are ≥ the PQL,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
If an initial dilution was performed on any sample, all surrogate recoveries are <10%, and all results are non-detect,	qualify all associated sample results as "R."
If there are two or more analyses for a particular fraction at the same dilution,	determine which analysis contains the best data to report using the considerations below, qualify all data from the rejected analysis as "R," and document the reason for rejecting data from one analysis in the data validation report. Considerations should include: 1. surrogate recovery (marginal vs. gross deviation); 2. holding times; 3. comparison of the values of the target analytes reported in each fraction; and 4. performance of ISs.
For surrogate recoveries out of specification, the following approaches are suggested based on a review of all data from the batch, especially considering the apparent complexity of the sample matrix: if at least two surrogates in the base/neutral fraction or the acid fraction, or one surrogate in the volatile fraction, are out of specification low but have recoveries ≥ 10%,	qualify all detects for that fraction as "J-" and all non-detects for that fraction as "UJ."

Evaluation (concluded)	Action (concluded)
if any surrogate recovery in a fraction is <10%,	qualify all detects for that fraction as "J-" and all non-detects for that fraction as "R."
if at least two surrogates in the base/neutral or the acid fraction, or one surrogate in the volatile fraction, are out of specification high,	qualify all detects for that fraction as "J+."

Criteria: In the case of a blank analysis with surrogates out of specification, special consideration must be given to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process.

If one or more samples in the batch show acceptable surrogate recoveries, the blank problem may be considered an isolated occurrence. However, even if this judgment allows some use of the affected data, analytical problems remain that must be corrected by the laboratory.

Evaluation	Action
If surrogate recovery in the blank does not meet acceptance criteria,	all detects < the PQL in all samples associated with the blank may be qualified as "J" and all non-detects in all samples associated with the blank may be qualified as "UJ."

4.2.6 IS Performance

IS criteria ensure that GC/MS sensitivity and response are stable and acceptable during each analysis.

Criteria: Sample and blank IS results must be within limits given in the specific SW-846 method.

IS area counts must not vary by more than a factor of two (50% to 200%) from the average of those obtained from the calibration standards.

The retention time (RT) of the IS must not vary more than ±30 seconds from that of the associated calibration standard.

When qualification of sample results is warranted due to failure of an IS to meet RT or area count acceptance criteria, results of all target compounds associated with that IS are qualified.

Refer to Appendix E for IS/target compound correlation guidelines.

Evaluation	Action
If there are two analyses for a particular fraction,	determine which analysis contains the best data to report using the considerations below, qualify all data from the rejected analysis as "R," and document the reason for rejecting the data from one analysis in the data validation report. Considerations should include: 1. magnitude of the RT shift; 2. holding times; 3. comparison of the values of the target compounds reported in each fraction; and 4. surrogate recovery.
If any IS area count is <50% of the average of that obtained from the calibration standards,	qualify all associated detects as "J+" and all associated non-detects as "UJ." Non-detects may be qualified as "R" based on professional judgment if the IS area counts are <25% of that of the average obtained from the calibration standards. **Note:** If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated.
If the IS area count is >200% of the average of that obtained from the calibration standards,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
If the IS RT varies by more than ±30 seconds from that of the associated CCV standard,	qualify all associated detects as "N" or "R" and all associated non-detects as "R."

4.2.7 MS/MSD

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on samples of various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

Criteria: The MS/MSD data shall not be used to qualify field sample results unless the MS/MSD sample was from the same client and of similar matrix.

An MS and MSD sample shall be analyzed at a frequency of once per data package, once per 20 samples of similar matrix, or once per sample matrix, whichever is more frequent.

The laboratory shall not use FBs, EBs, or TBs to satisfy these requirements, if the laboratory can identify these blanks.

Unless otherwise stated in the specific method, the MS and MSD accuracy and precision acceptance criteria shall be those calculated by the laboratory using the procedure given in SW-846 Method 8000C. If the acceptance criteria are not given, recovery limits of 70% to 130% and ±30% RPD should be used as the criteria. It may be appropriate to use wider default recovery acceptance criteria for SVOC analysis based on professional judgment. For solid and waste samples, it may be appropriate to accept up to a 40% RPD based on the professional judgment. The MS and MSD %R must be within the acceptance limits, unless the sample concentration is > 4X the spike concentration (see Section 4.1.20).

The MS and MSD analyses must meet all sample analysis acceptance criteria. An effort to determine to what extent the results of the MS/MSD affect the associated data should be made. This determination should be made considering the MS/MSD sample matrix, the surrogate recoveries, and the LCS results.

Professional judgment should be used to determine if MS/MSD failure warrants qualification of only the results for the failed compounds, or if results for all the compounds associated with the failed MS compound and its associated IS are affected. Generally, unless evidence exists to warrant qualification of other compounds, only the compounds in the MS spiking mixture shall be qualified.

For programs that require application of one final qualifier to sample results, if a recovery (accuracy) infraction is identified in one or both of the MS samples along with an RPD (precision) infraction between the MS and MSD, the sample is qualified for the accuracy infraction. For example, if a compound has low MS recovery and the RPD is not within criteria, the data are qualified as “J-.”

Evaluation	**Action**
If the MS/MSD analysis was from another client or of a dissimilar matrix; if the frequency of the MS/MSD did not meet specified criteria; if no MS/MSD was analyzed; or if FB, EB, or TB samples were used for MS/MSD purposes,	qualify all detects as “J” and all non-detects as “UJ.”

Evaluation (concluded)	**Action (concluded)**
If no other measure of precision (i.e., LCSD or replicate) is available,	qualify all detects as "J" and all non-detects as "UJ."
If the surrogate, IS, and LCS %Rs are within the required acceptance criteria and…	
either the MS or MSD %R for any target compound is > the upper acceptance limit,	qualify all associated detects as "J+."
either the MS or MSD %R for any target compound is < the lower acceptance limit,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the recovery is ≥10% and as "R" if the recovery is <10%.
If the RPD for any target compound does not meet the acceptance criteria or %Rs fail both high and low,	qualify all associated detects for that compound as "J" and all associated non-detects as "UJ."

Note: The laboratory may analyze TBs in a separate batch than that of soil samples due to differences in sample matrices. In this situation, the laboratory may not analyze an MS/MSD for the batch associated with the TBs. The TB results should then be assessed for accuracy and precision using an LCS/LCSD.

4.2.8 Replicate

Replicate analyses are indicators of laboratory precision based on each sample matrix. If a replicate was performed instead of an MSD, the following criteria are applied. If insufficient sample was submitted to analyze an MS/MSD or replicate, the laboratory may run a LCSD to measure precision. LCSD precision shall be assessed as described in Section 4.2.7.

Criteria: A replicate sample shall be analyzed at a frequency of once per data package, once per 20 samples of similar matrix, or once per sample matrix, whichever is more frequent. All sample acceptance criteria must be met in the replicate analysis.

Samples identified as FBs, EBs, or TBs should not be used for replicate sample analysis.

Unless otherwise stated in the specific method, the replicate precision acceptance criteria shall be those calculated by the laboratory using the procedure given in SW-846 Method 8000C. When no laboratory-derived control limits are reported, a control limit of 30% for the RPD shall be used for

sample values ≥5X the PQL. For solid and waste samples, it may be appropriate to accept up to a 40% RPD based on the professional judgment.

A control limit of ± the PQL shall be used for sample values < 5X the PQL, including the case when only one of the replicate sample values is <5X the PQL.

No precision criteria apply when both replicate sample values are < the PQL.

Evaluation	**Action**
If no replicate sample, no MSD, and no LCSD were analyzed for each matrix or for each data package,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."
If an FB, EB, or TB was used for the replicate analysis and no MSD or LCSD was run,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."
If the original result and replicate result for target compound are both ≥5X the PQL, and the RPD exceeds the appropriate control limit,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."
If the original and/or replicate result for any target compound is <5X the PQL (including non-detects) and the difference between the original result and replicate result is > the PQL,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."

4.2.9 LCS

Data for LCSs are generated to provide information on the accuracy of the analytical method and the overall laboratory performance, including sample preparation.

Criteria: An LCS should be analyzed for all methods at a frequency of once per data package, once per matrix, or once per 20 analytical samples, whichever is most frequent. The LCS should have recoveries for all target analytes; however, for very large analyte lists or for known poor performers, the laboratory may have received an exemption for one or more analytes.

The LCS must meet all sample acceptance criteria. If surrogate and IS acceptance criteria are not met in the LCS analysis, the LCS must be reanalyzed. The LCS should meet all method-specific LCS requirements and acceptance criteria. If the recovery acceptance criteria are not reported, the reviewer should use the criteria in Appendix F, or 70% to 130% as the criteria.

If the laboratory analyzed an LCS/LCSD as a measure of precision both the LCS and LCSD must meet recovery acceptance criteria.

General laboratory precision and accuracy can be evaluated using the LCS acceptance criteria and the interlaboratory comparison data given in Appendix F. Individual LCS recoveries may be evaluated against the criteria in Appendix F if the laboratory's criteria are significantly different from those in the tables.

For volatile organics in an aqueous matrix, a successful **second source** CCV meets the LCS requirements.

Evaluation	**Action**
If, based on professional judgment, the laboratory's internal acceptance criteria are excessively wide or acceptable recoveries are significantly biased,	notify the program manager.
If the frequency of the LCS did not meet the specified criteria,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If results are reported for target compounds that are not in the LCS,	detects for those compounds may be qualified as "J" and non-detects for those compounds may be qualified as "UJ" based on professional judgment. Compounds missing under an exemption may be qualified based on professional judgment.

If the LCS criteria were not met and reanalysis was not performed, then the laboratory performance and method accuracy are in question. Professional judgment should be used to determine if data should be qualified for all target compounds or just those compounds associated with the failed LCS compound and its associated IS. The following may be used as guidance in qualifying data.

If a full or large TAL LCS is analyzed, the following criteria may be used for LCS %Rs which fall outside reported acceptance criteria but are >10%:

70 to 74 compounds	$\leq$ 5 LCS fall outside acceptance criteria - no qualification
60 to 69 compounds	$\leq$ 4 LCS fall outside acceptance criteria - no qualification
50 to 59 compounds	$\leq$ 3 LCS fall outside acceptance criteria - no qualification
40 to 49 compounds	$\leq$ 2 LCS fall outside acceptance criteria - no qualification
30 to 39 compounds	$\leq$ 1 LCS fall outside acceptance criteria - no qualification
< 30 compounds	No LCS fall outside acceptance criteria - no qualification

Evaluation	Action
If the LCS %R is > the upper acceptance limit,	qualify all associated detects as "J+."
If the LCS %R is < the lower acceptance limit,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the %R is ≥10% and as "R" if %R is <10%.
If %Rs for more than half of the compounds in the LCS analysis are below the acceptance range,	qualify all detects as "J-" and all non-detects as "UJ" if the failures are marginally low and as "R" if %Rs are significantly below acceptance limits. **Note**: If recoveries of more than half of the compounds in the LCS analysis are below the acceptance range, the laboratory has not shown that it can actually meet program required DLs.
If %Rs for more than half of the compounds in the LCS analysis are above the acceptance range,	qualify all detects as "J+."
If %Rs for more than half of the compounds in the LCS analysis are outside the acceptance range, both above and below, or if an LCS/LCSD pair was analyzed and recoveries of any target compound are both above and below acceptance criteria,	qualify all detects in all associated samples as "J" and all non-detects in all associated samples as "UJ."

4.2.10 Sample Carry-over

Sample carry-over may occur when a high-concentration sample is analyzed immediately prior to another field sample. Steps must be taken to avoid introduction of false positive results in the second sample analysis due to instrument contamination.

Criteria: The absence of sample carry-over must be determined and verified. If examination of the run logs indicates that any samples in the analytical run of interest required dilution, and there is no documentation of a rinse or blank analysis immediately following the original undiluted analysis then sample carry-over may be suspected in the subsequent sample.

Evaluation	Action
If any target compound found in the sample requiring dilution exceeded the high calibration standard and was also found in the following sample at a concentration <5X the PQL,	qualify the result for that compound in the second sample as "R" or "NJ" based on professional judgment.
If no data are available for the sample that required dilution and the laboratory has not documented that carry-over was evaluated, and the compound was also found in the following sample at a concentration <5X the PQL,	qualify the results for that compound in the second sample as "N."

4.2.11 Dilutions

Criteria: The PQLs must be adjusted to reflect all sample dilutions, concentrations, splits, clean-up activities, and dry weight factors that are not accounted for by the method.

Samples must be diluted and reanalyzed when any analytes exceed the calibration range.

Data from original samples should be included when any sample requires dilution due to one or more compounds exceeding the calibration range.

The original undiluted results document the actual MDLs for non-detects.

Evaluation	Action
If the PQLs have not been properly adjusted,	request an amended report from the laboratory.
In some cases, initial dilutions are required because of expected high concentrations of non-target analytes or because one or more target analyte is expected to greatly exceed the instrument working range. In these instances, the laboratory may not be able to analyze the undiluted sample.	note the dilution and elevated MDLs in the data validation report.
If any target compound exceeds the calibration range and… the original undiluted sample result was reported,	 qualify all detects from the undiluted analysis that exceeded the calibration range as "J."

Evaluation (concluded)	Action (concluded)
the sample was diluted and reanalyzed, and the diluted sample data were reported, the original undiluted sample data were not provided,	qualify all non-detects from the diluted analysis as "UJ." request this information from the laboratory.
If data from the original sample run are unavailable,	refer to Section 4.2.5 for assessment of initially diluted samples with low surrogate recovery.

Criteria: The laboratory shall strive to make dilutions in such a way that the final concentration is measured in the mid-range of the calibration curve and that results are not reported from measurements below the lowest concentration standard.

Evaluation	Action
If the instrument response (reported result / dilution factor) from a diluted sample is < that of the lowest concentration standard,	qualify all associated detects from the diluted analysis as "J."

Criteria: The extraction efficiency for extremely high concentrations of analytes has generally not been determined for most methods. If the analysis requires an extraction and dilutions of >100,000:1 the efficiency of the extraction may be suspect.

Evaluation	Action
If dilutions of >100,000:1 was required,	qualify all associated detects as "J-."

4.2.12 Mass Spectra Acceptability

Mass spectra review is typically outside the scope of routine data validation and should not be performed unless it is specifically requested by the SMO. When mass spectra review is required by the program, it must be performed by a validator experienced in the interpretation of mass spectra.

The laboratory is to identify mass spectra using either the National Bureau of Standards (NBS) /EPA/Mass Spectrometry Data Centre (MSDC) library or the National Institute of Standards and Technology (NIST)/EPA/National Institutes of Health (NIH) library. The laboratory must identity and document peaks and reference spectra for all target compounds with concentrations above the MDL. While it is not the function of the validator to determine if the analyst correctly identified a

compound, an evaluation of how well the analyte peak matches the reference spectra may be requested. To evaluate analyte spectra, the guidelines in Appendix G shall be used.

Evaluation	**Action**
If the laboratory does not identify mass spectra using a nationally recognized standard library,	notify the program manager.
If the sample spectrum does not match the reference spectrum, the extracted ion current profiles (EICPs) RT or relative retention time (RRT) does not meet criteria, or several guideline failures were observed,	qualify all associated results as "R."
If the analyte is not identifiable due to gross interference or apparent instrument instability,	qualify all associated results as "R."
If the analyte was misidentified by the laboratory,	request an amended report from the laboratory.
If identification of the analyte was hampered by interferences such that it is not certain that a positive identification could be made,	qualify all associated results as "N" based on professional judgment or request additional data from the laboratory.

4.2.13 TICs

Chromatographic peaks that are not target analytes, surrogates, or ISs are potential TICs. TIC evaluation is typically outside the scope of routine data validation and should not be performed unless specifically requested by the SMO. When TIC evaluation is required by the program, it must be performed by validators with experience in mass spectra interpretation**.**

Criteria: For each sample, the laboratory may be requested to conduct a mass spectral search of either the NBS/EPA/MSDC library or the NIST/EPA/NIH library. The laboratory may report the possible identity for up to 20 of the largest VOC fraction peaks and the 20 largest SVOC fraction peaks which are not surrogate, IS, or target compounds, but which have an area/height >10% of the size of the area/height of the nearest IS.

It should be noted that common laboratory artifacts/contaminants and their sources (i.e., aldol products, solvent preservatives/reagent contaminants, etc.) may be present in blanks and not reported as sample TICs.

Examples:

- Common laboratory contaminants: CO_2 (mass/charge ratio (m/e) 44), siloxanes (m/e73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluoro-trichloromethane), phthalates at levels < 100 micrograms per liter (ug/L) or 4,000 micrograms per kilogram (ug/kg).

- Solvent preservatives: cyclohexene is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, chlorocyclohexanol.
- Aldo reaction products of acetone include 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, 5,5-dimethyl-2(5H)-furanone.

Evaluation	**Action**
If a low-level non-target compound that is a common artifact or laboratory contaminant is detected in a sample, If sample TIC results are not sufficiently above the level in the blank and the results are reported,	verify that TIC peaks present in samples are not found in blanks. Blank chromatograms should be examined for peaks that are <10% of the IS height but are present in the sample chromatogram at similar RRT. the results may be qualified as "R" (dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples).
If a result is identified as a TIC, If a compound is not found in any blanks, but is a suspected artifact or common laboratory contaminant,	qualify that result as "NJ." identify the compound as such in the data validation report. Compounds that are suspected artifacts or common laboratory contaminants result may be qualified as "R" based on professional judgment.

It should be noted that common laboratory calibration practices, along with limitations of some commercial software could result in compounds being detected and not reported in either the Form I or the TIC Summary Report. Review all quantitation reports to verify that all detected compounds are reported whenever a TIC Summary is included.

Evaluation	**Action**
If a compound is identified on the quantitation report but are not reported as target detect or as a TIC,	request a corrected report from the laboratory.

4.2.14 Method-specific Analytical Requirements–Organic GC/MS

The additional analytical requirements addressed below are organized by SW-846 Method. These requirements should be checked if the level of deliverable (level III or level IV) allows.

4.2.14.1 Method 8260B or 8260C, VOC Analysis by GC/MS

Criteria: The analysis of 2-chloroethyl vinyl ether in water must be performed on an unacidified sample.

Evaluation	Action
If 2-chloroethyl vinyl ether was reported for an acidified water sample,	qualify all associated detects as "NJ-" and all associated non-detects as "R."

4.2.14.2 Method 8270C or 8270D, SVOC Analysis by GC/MS

Criteria: Gel permeation chromatography (GPC) cleanup shall be used as necessary to eliminate interferences. In addition, all water samples containing high molecular weight compounds that interfere with the analysis of the target compounds must also undergo GPC cleanup.

Evaluation	Action
If the runlog notations, spectral data, IS %Rs, or surrogate %Rs indicate potential interferences,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If appropriate extract cleanup was not performed,	note this on the data validation report.

4.2.14.3 Method 8280B, Polychlorinated Dioxins and Furans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)

Sample analysis shall be performed according to the requirements listed in SW-846 Method 8280B. Evaluation of tuning reports is not required for this method.

Criteria: Initial calibration shall be performed using the five calibration solutions listed in Table 1 of the method. The %RSD for the ISs and the target compounds for the five calibration standards must be <15%.

Calibration verification shall be performed using the standards solution given in Table 4 of the method. The calibration verification analysis must meet the criteria given in Section 7.13.3.6 of the method.

Evaluation	Action
If the %RSD is >15% for any IS or target compound,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Evaluation (concluded)	Action (concluded)
If the CCV acceptance criteria were not met for any target compound,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Sample Analysis

Criteria: For identification of any compound, the ion abundance ratios must be within the limits specified in Table 9 of the method.

For 2,3,7,8-substituted compounds that have an isotopically labeled IS or recovery standard present in the sample extract, the RT must be –1 to +3 seconds of the isotopically labeled standard. For 2,3,7,8-subtituted compounds that do not have an isotopically labeled IS or recovery standard present in the sample extract, the RT must fall within 0.005 RRT units of the RRT measured in the continuing calibration.

For non-2,3,7,8-substituted compounds, the RT must be within the corresponding homologous RT windows established by analyzing the column performance check solution.

Evaluation	Action
If ion abundance ratio criteria were not met for any compound,	qualify all associated results as "R."
If the RT of any compound is outside of the RT window,	qualify all associated results as "R."

Criteria: IS %R for analytical samples must be ≥25% and ≤150%. IS recovery guidelines are discussed in Section 7.15.5 of the method.

The LCS shall contain all of the target compounds at concentrations near the midpoint of the calibration range.

Evaluation	Action
If the recovery of any IS solution compound is >150%,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
If the recovery of any IS solution compound is <25%,	qualify all associated detects as "J+" and all associated non-detects as "UJ" if the recovery is ≥10% and as "R" if the recovery is <10%

Evaluation (concluded)	Action (concluded)
If results are reported for target compounds that are not in the LCS,	qualify all associated detects as "J" and all associated non-detects as "UJ."

GC Column Performance

Criteria: The GC column performance solution is used for defining the homologous GC RT windows and to document the chromatographic resolution. Column performance must be evaluated at the beginning of each 12-hour analytical period and must meet method acceptance criteria (see Section 7.12 of the method) before sample analysis may begin.

Evaluation	Action
If GC column performance was not evaluated at the required frequency or if method criteria were not met,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Confirmation of 2,3,7,8-Tetrachlorodibenzofuran (TCDF) Detects

Criteria: The DB-5 GC column generally used for polychlorinated dibenzodioxin (PCDD) and polychlorinated dibenzofuran (PCDF) analyses does not adequately separate 2,3,7,8-TCDF from its closest eluting isomer. If 2,3,7,8-TCDF is detected in a sample, the result must be confirmed on a second column capable of separating 2,3,7,8-TCDF from all other TCDF homologues (as proven by successful analysis of the GC column performance mix with <25% valley between 2,3,7,8-TCDF and its closest eluting isomer).

Evaluation	Action
If 2,3,7,8-TCDF was detected in a sample and the result was not confirmed on a second column with successful analysis of the GC column performance mix,	qualify all associated detects as "NJ."

4.2.14.4 Method 8290A, PCDD and PCDF by HRGC/High Resolution Mass Spectrometry (HRMS)

Initial Calibration

Criteria: A 5-point calibration is prepared for each labeled and unlabeled compound. The relative response factor (RRF) %RSD for the unlabeled standards must be

≤20%. For the labeled compounds, the %RSD must be ≤30%. Ion abundance ratios must meet the criteria listed in Table 8 of the method.

Evaluation	**Action**
If the %RSD is:	
>20% for any unlabeled calibration standard or >30% for any labeled calibration standard, but ≤ 40%,	qualify all associated detects as "J" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>40% but ≤60% for either a labeled or unlabeled calibration standard,	qualify all associated detects as "J" and all associated non-detects as "UJ."
>60% for either a labeled or unlabeled calibration standard,	qualify all associated detects as "J" and all associated non-detects as "R."
If the ion abundance criteria is not met for any compound,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Continuing Calibration

Criteria: Calibration must be verified for both unlabeled and labeled compounds at the beginning and end of each 12-hour shift during which analysis is performed.

The measured RFs must be ≤20% of the mean values established during ICAL for unlabeled compounds and ≤30% of the mean values established during ICAL for labeled compounds. The ion abundance must be within the limits in Table 8 of the method.

For the calibration verification analyzed at the beginning of a 12-hour shift, the effect on data quality of a standard that does not meet criteria must be assessed using professional judgment. Guidance is provided in Section 7.7.4.4 of the method. For the calibration verification analyzed at the end of a 12-hour shift, a %D of 25% for unlabeled compounds and 35% for labeled compounds is acceptable; however, in this instance, the mean RFs from the beginning and ending daily calibration runs are used to calculate analyte concentrations instead of the RFs obtained from the ICAL. If the %D of the ending calibration is >25% for any unlabeled compound and/or >35% for any labeled compound, then successful performance of another ICAL must be analyzed within two hours of sample analysis for the data to be acceptable. In this case, the mean RFs from the beginning and ending daily calibration runs are still used to calculate analyte concentrations.

Evaluation	Action
If the ion abundance ratio for any compound is outside of the method limits,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If the %D criteria are not met for any CCV compound at the beginning of a 12-hour shift, and… the %D is positive, the %D is negative,	 qualify all associated detects as "J+." . qualify all associated detects as "J-" and if any other calibration criteria have been exceeded for that compound, qualify all associated as "UJ."
If the %D criteria were <u>not</u> met for any compound at the end of a 12-hour shift, a new ICAL was analyzed within two hours of sample analysis, and… the %D is positive, the %D is negative,	 qualify all associated detects as "J+." qualify all associated detects as "J-" if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
If the %D criteria were not met for any compound at the end of a 12-hour shift and a new ICAL was <u>not</u> analyzed within two hours of sample analysis,	qualify all sample data analyzed during that 12-hour shift as "R."

Sample Preparation

Criteria: Extract cleanup shall be performed to eliminate interferences. The laboratory shall first partition the sample extract, followed by silica/alumina column cleanup and carbon column cleanup.

Evaluation	Action
If the documentation on the run log, spectra data, and/or IS or labeled compound %Rs indicate interferences and extract cleanup was not performed,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Sample Analysis

Criteria: For identification of any compound, the ion abundance ratios must be within the limits specified in Table 8 of the method.

For 2,3,7,8-substituted compounds which have an isotopically labeled IS or recovery standard present in the sample extract, the RT must be -1 to +3 seconds of the isotopically labeled standard. For 2,3,7,8-substituted compounds that do not have an isotopically labeled IS or recovery standard present in the sample extract, the RT must fall within 0.005 RRT units of the RRT measured in the continuing calibration.

For non-2,3,7,8-substituted compounds, the RT must be within the corresponding homologous RT windows established by analyzing the column performance check solution.

Evaluation	Action
If ion abundance ratio criteria are not met for any compound,	qualify all associated results as "R."
If the RT of any compound is outside of the RT window,	qualify all associated results as "R."

Mass Spectrometer Performance Criteria

Criteria: Performance criteria are established to ensure mass resolution, identification and, to some degree, sensitivity. These criteria are not sample specific.

Conformance is determined using standard materials. These criteria should be met in all circumstances. Mass spectrometer performance must be checked at the beginning and end of each analytical period in accordance with the method criteria (see Section 8.2 of the method).

Evaluation	Action
If mass spectrometer performance was not checked at the required frequency or if method criteria were not met,	qualify all associated detects as "R" and all associated non-detects as "UJ."

Replicate Samples

Criteria: A replicate sample should be extracted and analyzed with each batch of samples. The RPDs between results (i.e., between the recoveries for the labeled 2,3,7,8-substituted compounds and between the concentrations for the non-labeled 2,3,7,8-substituted compounds) should be ≤25%.

Note: An MS/MSD is not required for this method since it is an isotope dilution analysis. A replicate sample or LCS/LCSD will suffice to demonstrate batch precision.

Evaluation	Action
If a replicate sample or LCSD were not analyzed for each matrix or for each data package,	qualify all detects of the same matrix as "J" and all non-detects of the same matrix as "UJ."
If the RPD between the sample (or LCS) and its replicate (or LCSD) for any compound falls outside the appropriate control window,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."

ISs

Criteria: The laboratory must spike all samples with the sample fortification solution and all sample extracts with recovery standard solution. The %R of each compound must be within 40% to 135%.

Evaluation	Action
If the %R for any sample fortification solution compound is <40%,,	qualify all detects for that sample fraction as "J+" and all non-detects for that sample fraction as "UJ" if the %R is ≥10% and as "R" if the %R is <10%.
If the %R for any sample fortification solution compound is >135%,	qualify all detects for that sample fraction as "J-" and all non-detects for that sample fraction as "UJ."

Gas Chromatography (GC) Column Performance

Criteria: The GC column performance solution is used for defining the homologous GC RT windows and to document the chromatographic resolution. Column performance must be checked at the beginning of each analytical analysis period and must meet method acceptance criteria (see Section 8.2 of the method) before sample analysis may begin.

Evaluation	Action
If GC column performance is not checked at the required frequency or if method criteria is not met,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Confirmation of 2,3,7,8-TCDF Detects

Criteria: The DB-5 GC column generally used for PCDD and PCDF analyses does not adequately separate 2,3,7,8-TCDF from its closest eluting isomer. If 2,3,7,8-TCDF is detected in a sample, the result must be confirmed on a second column capable of separating 2,3,7,8-TCDF from all other TCDF homologues (as proven by successful analysis of the GC column performance column mix with <25% valley between 2,3,7,8-TCDF and its closest eluting isomer).

Evaluation	Action
If 2,3,7,8-TCDF was detected in a sample and the result was not confirmed on a second column with successful analysis of the GC column performance mix,	qualify all associated detects as "NJ."

4.2.14.5 Method TO-14A and TO-15, VOCs in Ambient Air using GC/MS

Analysis shall be performed according to the requirements specified in EPA Method TO-14A, *"Determination of VOCs in Ambient Air Using SUMMA® Passivated Canister Sampling and Gas Chromatographic Analysis,"* Revision 1.0 or TO-15, "*Determination of VOCs in Air Collected in Specially Prepared Canisters and Analyzed by GC/MS."* In general, validate these analyses according to Section 4.2.

Surrogates, an MS/MSD, and TICs are not required for these methods.

MDLs are not used for TO-15, detects are only reported above the PQL.

Instrument Tuning for GC/MS

See Section 4.2.1 for tuning and performance criteria.

Initial Calibration

Criteria: Instrument calibration shall be performed using at least three standard concentration levels (five standards for TO-15) and a humid zero air standard (not required for TO-15). In addition, a zero air certification for the sampling apparatus is to be provided.

Evaluation	Action
If an insufficient number of standards were used,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Evaluation (concluded)	Action (concluded)
If a zero air certification is not provided,	document the occurrence in the data validation report

RFs

Criteria: The %RSD for the RFs must be ≤30% and the average RF shall be ≥ the method-specified minimum RF for each compound. Compounds without specified minimum RFs will be >0.05.

Evaluation	Action
If the average RF for any target compound is < the specified minimum RF, or <0.05 if no minimum is specified,	qualify all associated detects as "J" and all associated non-detects as "UJ" if the average RF is ≥0.01 and as "R" if the average RF is <0.01.
If the %RSD for any target compound is...	
≤30%,	qualify all associated detects as "J" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>45% but ≤60%,	qualify all associated detects as "J" and all associated non-detects as "UJ."
>60%,	qualify all associated detects as "J" and all associated non-detects as "R."

Calibration Verification

Criteria: Prior to analysis of samples, a calibration standard must be analyzed immediately following an ICAL to ensure that the instrument continues to remain under control.

A calibration standard must be analyzed:

(1) daily and

(2) contain all target compounds.

The laboratory is allowed to perform corrective action and reanalyze once after a failure. If more than two calibration standards were analyzed to obtain a passing calibration standard, then the calibration was not verified and the calibration verification frequency criteria was not met.

Evaluation	Action
If the calibration standard was not analyzed at the proper frequency, or if all target compounds were not present in any calibration standard,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If the required calibration standard was not analyzed,	qualify all associated detects as "J" and all associated non-detects as "R."

RFs

Criteria: The %D between RFs and the average RFs obtained from the ICAL shall be calculated according to the formula in Section 6.3 and must be ≤30%.

Evaluation	Action
If the %D was reported with the wrong sign (e.g., + %D for a negative bias),	document the occurrence in the data validation report and assess any infractions using the correct sign.
If the %D between an ICAL RF and continuing calibration RF for any target compound is...	
>30% and positive (high bias),	qualify all associated detects as "J+."
>30% but ≤45% and negative (low bias),	qualify all associated detects as "J-" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>45% but ≤60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
>60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "R."

Blanks

Criteria: A daily humid zero air instrument blank (not required for TO-15) shall be analyzed immediately prior to and after instrument calibration. These instrument blank results must be <0.2 parts per billion by volume (ppbv) in all target analytes before analysis may proceed.

Evaluation	Action
If a humid zero air instrument blank was not analyzed at the required frequency,	qualify all associated detects <5X the PQL as "J."
If any target compound was detected in the instrument blank at a level ≥ the MDL but <0.2 ppbv,	qualify all results as discussed in Section 4.2.4.

IS Performance

Criteria: IS area counts must not vary by more than ±40% from the average of those obtained from the calibration standards.

The RT of the IS must not vary more than ±0.33 minutes (20 sec.) from that of the associated calibration standard.

When qualification of sample results is warranted due to failure of an IS to meet RT or area count acceptance criteria, results of all target compounds associated with that IS are qualified.

Refer to Appendix E for IS/target compound correlation guidelines.

Evaluating previous CCV IS areas are not required for this method.

Evaluation	Action
If there are two analyses for a particular fraction,	determine which analysis contains the best data to report using the considerations below, qualify all data from the rejected analysis as "R," and document the reason for rejecting the data from one analysis in the data validation report. Considerations should include: 1. magnitude of the RT shift; 2. holding times; 3. comparison of the values of the target compounds reported in each fraction.
If any IS area count is <40% of the average of that obtained from the calibration standards,	qualify all associated detects as "J+" and all associated non-detects as "UJ." Non-detects may be qualified as "R" based on professional judgment if the IS area counts are <20% of that of the average obtained from the calibration standards.

Evaluation (concluded)	Action (concluded)
	Note: If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated.
If the IS area count is >140% of the average of that obtained from the calibration standards,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
If the IS RT varies by more than ±0.33 minutes from that of the associated calibration standard,	qualify all associated detects as "N" or "R" and all associated non-detects as "R."

LCS/LCSD

See Section 4.2.9 for LCS/LCSD criteria.

4.2.14.6 Method 1668A, Chlorinated Biphenyl Congeners

Initial Calibration

Criteria: Isotope dilution shall be used for calibration of the toxics and beginning and ending level of chlorination (LOC) chlorinated biphenyls (CBs). A 5- or 6-point calibration is prepared for each native congener. The RRF %RSD for any native toxics/LOC CBs must be <20%. If a linear curve is used for ICAL, the r^2 of the curve must be >0.99.

Evaluation	Action
If the %RSD for any target compound is…	
>20% but ≤40%,	qualify all associated detects as "J" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>40% but ≤60%,	qualify all associated detects as "J" and all associated non-detects as "UJ."
>60%,	qualify all associated detects as "J" and all associated non-detects as "R."

Evaluation (concluded)	Action (concluded)
If the r^2 for any target compound is…	
<0.99 but ≥0.90,	qualify all associated detects as "J" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
<0.90 but ≥0.80,	qualify all associated detects as "J" and all associated non-detects as "UJ."
<0.80,	qualify all associated detects as "J" and all associated non-detects as "R."

Criteria: Calibration using ISs is used for determination of native CBs for which a labeled compound is not available. For these CBs, calibration is performed at a single point. Compounds should be quantitated using the appropriate reference IS listed in Table 2 of the method. Ion abundance ratios must meet the criteria in Table 8 of the method or must be within 15% of the theoretical ratio of the ion monitored.

Evaluation	Action
If the ion abundance criteria were not met for any calibration compound,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Continuing Calibration

Criteria: At the beginning of each 12-hour shift during which analyses are performed, calibration is verified for all native CBs and labeled compounds. The ion abundance ratios for all CBs must be within the limits in Table 8 and all compounds must meet the calibration verification recovery limits listed in Table 6 of the method.

RRTs of native CBs and labeled compounds in the calibration verification must be within ± 0.5% of the mean RRT determined in the ICAL or most recent calibration verification standard. The diluted combined 209-congener solution must be analyzed as a final step in the calibration verification and must meet minimum analysis and resolution specifications of the method.

Evaluation	Action
If the ion abundance ratio for any compound is outside of the method limits,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If the verification limits are not met for any calibration verification compound and… the %R is above the verification limits, the %R is below the verification limits,	 qualify all associated detects as "J+." qualify all associated detects as "J-" and all associated non-detects as "UJ" if the recovery is ≥10% and as "R" if the recovery is <10% .
If the RRT of any compound is outside of the RRT window,	qualify all associated results as "R."

RT Calibration

Criteria: The absolute RT of CB 209 must be ≥55 minutes if the SPB-octyl column is used. If a GC column or column system alternate to the SPB-octyl column is used, the absolute RT of CB 209 must be ≥ the laboratory-established minimum RT for CB 209. If the laboratory has not established a minimum RT value for CB 209, the RT for CB 209 must be ≥55 minutes.

Evaluation	Action
If an SPB-octyl column was used, and the absolute RT of CB 209 is <55 minutes,	qualify all associated results as "R."
If a GC column or column system alternate to the SPB-octyl column was used and the absolute RT is < the laboratory-established minimum RT for CB 209, or <55 minutes if the laboratory has not established a minimum RT,	qualify all associated results as "R."

Ongoing Precision and Recovery (OPR)

Criteria: OPR must be established for every batch of samples extracted and analyzed and must meet the recovery and %RSD limits listed in Table 6 of the method. If the OPR criteria are not met and reanalysis was not performed, then the laboratory performance and method accuracy are in question.

Evaluation	Action
If the frequency of the OPR did not meet the specified criteria,	note the deficiency in the data validation report.
If the OPR %R is > the upper acceptance limit,	qualify all associated detects as "J+."
If the OPR %R is < the lower acceptance limit,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the %R is ≥ 10% and as "R" if the %R is < 10%.
If %Rs for more than half of the compounds in the OPR analysis are below the acceptance range,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the failures are marginally low and as "R" if %Rs are significantly below acceptance limits. **Note**: If recoveries for more than half of the compounds in the OPR analysis are below the acceptance range, the laboratory has not shown that it can actually meet program required DLs.
If %Rs for more than half of the compounds in the OPR analysis are above the acceptance range,	qualify all associated detects as "J+."
If %Rs for more than half of the compounds in the OPR analysis are outside the acceptance range, both above and below,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Sample Preparation

Criteria: CBs may be bound to suspended particles in aqueous samples; therefore, the preparation of aqueous samples is dependent upon the solids content of the sample. A direct extraction is used for aqueous samples containing <1% solids. For aqueous samples containing >1% solids, the sample is agitated, allowed to settle and the liquid is decanted and discarded prior to extraction of the solids. The particle size for all solid samples should be determined prior to preparation. Particle size must be 1 millimeter or less prior to sample preparation.

Evaluation	Action
If % solids and particle size were not determined prior to sample preparation or if the proper preparation method was not performed,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Criteria: Extract cleanup shall be used as necessary to eliminate interferences. The laboratory may employ GPC, acid, neutral, or base silica gel; florisil; carbopak/celite; or high-performance liquid chromatography (HPLC) cleanup methods or anthropogenic isolation column for lipids (tissue extracts only).

Evaluation	Action
If the documentation on the run log, spectra data, and/or IS or labeled compound recoveries indicate interferences and applicable cleanup was not performed,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Sample Analysis

Criteria: For identification of any CB or labeled compound, the ion abundance ratios must be within the limits specified in Table 8 of the method or ±15% of the calibration verification standard. The RRT of each CB must be within ±0.5% of the mean RRT determined in the ICAL or ±0.5% of the RRT from the most recent calibration verification standard.

Evaluation	Action
If ion abundance ratio criteria are not met for any compound,	qualify all associated results as "R."
If the RRT of any CB is outside of the RRT window,	qualify all associated results as "R."

Mass Spectrometer Performance Criteria

Criteria: Performance criteria are established to ensure mass resolution, identification, and, to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials. These criteria should be met in all circumstances.

Evaluation	Action
If mass spectrometer performance was not checked at the required frequency or if method criteria were not met,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Labeled Compounds

Criteria: To assess method performance on the sample matrix, the laboratory must spike all samples with the labeled toxics/LOC/window defining standard spiking solution and all sample extracts with the labeled cleanup standard spiking solution. The recovery of each labeled compound must be within the limits listed in Table 6 of the method.

Evaluation	Action
If the %R for any labeled toxics/LOC/window defining standard compound is below acceptance limits,	qualify all detects for that sample fraction as "J+" and all non-detects for that sample fraction as UJ" if the recovery is ≥10% and as "R if the recovery is <10%.
If the %R for any labeled toxics/LOC/window defining standard compound is above acceptance limits,	qualify all detects for that sample fraction as "J-" and all non-detects for that sample fraction as "UJ."
If the %R for any labeled cleanup standard compound is below acceptance limits,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the recovery is ≥10% and as "R" if the recovery is <10%.
If the %R for any labeled cleanup standard compound are above acceptance limits,	qualify all associated detects as "J+."

4.2.14.7 California Environmental Protection Agency Air Resources Board; Method 428, PCDD, PCDF, and Polychlorinated Biphenyl (PCB) Emissions from Stationary Sources

Initial Calibration

Criteria: A 5-point calibration is prepared for each compound (see Tables 3, 5, and 10 of the method for standard concentrations). The RRF RSD for any compound must be ≤15%.

Evaluation	Action
If the %RSD for any compound is…	
>15% but ≤40%,	qualify all associated detects as "J" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>40% but ≤60%,	qualify all associated detects as "J" and all associated non-detects as "UJ."
>60%,	qualify all associated detects as "J" and all associated non-detects as "R."

Continuing Calibration

Criteria: At the beginning and end of each 12-hour shift during which analyses are performed, calibration is verified for all compounds. The measured RFs must be ≤30% of the mean values established during ICAL. The relative abundance must meet the requirements specified in Tables 7 and 13 of the method.

Evaluation	Action
If the mass ratio for any compound is outside of the method limits,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If the %D criteria were not met for any compound and…	
the %D is positive,	qualify all associated detects for that compound as "J+."
the %D is negative,	qualify all associated detects as "J-" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."

GC Column Performance

Criteria: The GC column performance solution is used for defining the homologous GC RT windows, to document the chromatographic resolution, and to check relative ion abundance criteria. Column performance must be checked at the beginning and end of each 12-hour analysis period and must meet method acceptance criteria (see Sections 5.3.5 and 6.3.5 of the method). If the laboratory operates during consecutive 12-hour shifts, analysis of the performance check solution at the beginning of each 12-hour period and at the end of the final 12-hour period is sufficient.

Evaluation	Action
If GC column performance was not checked at the required frequency or if method criteria is not met,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Sample Preparation

Criteria: Extract cleanup shall be performed to eliminate interferences. The laboratory shall first partition the sample extract and then follow with an appropriate cleanup procedure.

Evaluation	Action
If sample spectra and/or IS and/or surrogate recoveries indicate interferences and documentation of extract cleanup was not provided,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Sample Analysis

Criteria: For identification of any compound, the mass ratios must be within ±15% of the mass ratios listed in Tables 7 and 13 of the method. The RRT of each compound must be within ±0.006 RRT units of the standard RRT.

Evaluation	Action
If mass ratio criteria are not met for any compound,	qualify all associated results as "R."
If the RRT of any compound is outside of the RRT window,	qualify all associated results as "R."

ISs

Criteria: To assess method performance on the sample matrix, the laboratory must spike all samples with known concentrations of stable isotopically labeled ISs prior to extraction.

The laboratory must spike all samples with known concentrations of recovery ISs prior to injection. The %R of each IS must be within 40% to 120% of the known value and the absolute RTs must be within ±10 seconds of those measured during the last previous continuing calibration check.

If IS %Rs are outside of the acceptable limits, the signal to noise ratio of the IS must be $\geq$10.

Evaluation	Action
If the %R for any IS compound is below acceptance limits,	qualify all associated detects for that sample fraction as "J+" and all associated non-detects for that sample fraction as "UJ" if the recovery is ≥10% and as "R" if the recovery is <10%.
If the %R for any IS compound is above acceptance limits,	qualify all associated detects for that sample fraction as "J-" and all associated non-detects for that sample fraction as "UJ."

Matrix Blank

Criteria: Portions of the sample matrix (resin and filter) shall be analyzed at a frequency of every extraction set of 20 or fewer samples. All samples must be associated with an uncontaminated matrix blank. An uncontaminated matrix blank is defined as not having any compounds detected at a concentration ≥ the MDL. The sample results must not be corrected by subtracting blank values.

Matrix blanks should be evaluated in the same manner as an MB. Blank qualification guidelines are discussed in Section 4.2.4.

Blank Sampling Train

Criteria: There shall be a least one blank train submitted to the laboratory for each series of three or fewer test runs. For sources with air pollution control devices, there shall be at least one blank train assembled at the inlet, and one at the outlet of the air pollution control devices for each set of three or fewer runs at each location. All samples must be associated with an uncontaminated blank train. An uncontaminated blank train is defined as not having any compound detected at a concentration ≥ the MDL. The sample results must not be corrected by subtracting blank values.

Blank sampling trains should be evaluated in the same manner as an MB. Blank qualification guidelines are discussed in Section 4.2.4.

LCS

Criteria: An LCS must be extracted and analyzed with every batch of 20 samples or less and it must contain at least one representative of each chlorinated class of compounds to be determined in the samples. Accuracy is considered acceptable if the %R is within 60% to 140%.

Note: If the LCS criteria are not met and reanalysis was not performed, then the lab performance and method accuracy are in question.

Evaluation	**Action**
If the frequency of the LCS did not meet the specified criteria,	note the deficiency in the data validation report.
If there was not at least one compound associated with each chlorinated class of compounds,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If the %R for an LCS compound is >140%,	qualify all associated detects as "J+."
If the %R for an LCS compound is <60%,	qualify all associated detects as "J-", and all associated non-detects as "UJ" if the %R is ≥10% and as "R" if the %R is <10%.
If %Rs for more than half of the compounds in the LCS analysis are below the acceptance range,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the failures are marginally low and as "R" if %Rs are significantly below acceptance limits. **Note**: If %Rs for more than half of the compounds in the LCS analysis are below the acceptance range, the laboratory has not shown that it can actually meet program required DLs.
If %Rs for more than half of the compounds in the LCS analysis are above the acceptance range,	qualify all associated detects as "J+."
If %Rs for more than half of the compounds in the LCS analysis are outside the acceptance range, both above and below,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Mass Spectrometer Performance Criteria

Performance criteria are established to ensure mass resolution, identification and, to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials. These criteria should be met in all circumstances.

Criteria: Mass spectrometer performance must be checked every 12 hours of analysis in accordance with the method criteria. All compounds in all ICAL and continuing calibration standards must be within the QC limits listed in Tables 7 and 13 of the method for their respective isotopic ratios.

Evaluation	Action
If mass spectrometer performance was not checked at the required frequency or if method criteria are not met,	qualify all associated detects as "R" and all associated non-detects as "UJ."

QC Check Sample

Criteria: A QC check sample must be extracted and analyzed with every batch of 20 samples or less. Accuracy is considered acceptable if the %R is within 60% to 140% and precision is acceptable if the RPD is ≤30%.

Note: If the QC check sample criteria are not met and reanalysis was not performed, then the lab performance and method accuracy are in question.

Evaluation	Action
If the frequency of the QC check sample did not meet the specified criteria,	note the deficiency in the data validation report.
If any QC check sample RPD is > 30%,	qualify all associated detects of the same matrix as "J" and all associated non-detects as "UJ."
If the QC check sample %R is >140%,	qualify all associated detects as "J+."
If the QC check sample %R is <60%,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the %R is ≥10% and as "R" if the %R is <10%.
If %Rs for more than half of the compounds in the QC check sample analysis are below the acceptance range,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the failures are marginally low and as "R" if %Rs are significantly below acceptance limits.

Evaluation (concluded)	Action (concluded)
	Note: If %Rs for more than half of the compounds in the QC check sample analysis are below the acceptance range, the laboratory has not shown that it can actually meet program required DLs.
If %Rs for more than half of the compounds in the QC check sample analysis are above the acceptance range,	qualify all associated detects as "J+."
If %Rs for more than half of the compounds in the QC check sample analysis are outside the acceptance range, both above and below,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Spiked Sampling Trains

Criteria: Surrogate standards must be spiked into each sampling train as a means of estimating the precision and accuracy of the sampling train for collecting and recovering PCDDs, PCDFs, and PCBs in the stack gas sample. Surrogate recovery is considered acceptable if the %R is within 60% to 140%.

Evaluation	Action
If the surrogate %R is >140%,	qualify all associated detects as "J+."
If the surrogate %R is <60%,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the %R is ≥10% and as "R" if the %R is <10%.

4.2.14.8 Method 1613B, Tetra- through Octa-Chlorinated Dioxins and Furans by HRGC/HRMS

Note: An MS/MSD analysis is not required for this method.

Initial Calibration

Criteria: A combined 5-point calibration is prepared for the 2,3,7,8-substituted PCDDs and PCDFs for which labeled compounds are added to the samples (isotope dilution) and for 1,2,3,7,8,9-HxCDD, OCDF, and any non-2,3,7,8-

substituted compounds (ISs). The RRF %RSD for the compounds calibrated using isotope dilution must be ≤20%. For the compounds calibrated using ISs, the %RSD must be ≤35%. Ion abundance ratios must meet the criteria listed in Table 9 of the method.

The laboratory may use alternative ions for quantitation to eliminate interferences. In this case, the ion abundance ratios must meet the criteria set by the laboratory.

Evaluation	**Action**
If the %RSD is...	
>20% for any compound calibrated by isotope dilution, or >35% for any compound calibrated by IS, but ≤40%,	qualify all associated detects as "J" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>40% but ≤60% for any compound,	qualify all associated detects as "J" and all associated non-detects as "UJ."
>60% for any compound,	qualify all associated detects as "J" and all associated non-detects as "R."
If the ion abundance criteria were not met for any compound,	qualify all associated results as "R."

Continuing Calibration

Criteria: At the beginning of each 12-hour period during which analysis is performed, calibration is verified for all compounds. The measured concentration of each compound must be within the limits set in Table 6 of the method. The ion abundance must be within the limits in Table 9 of the method.

The absolute RTs of the ^{13}C-1,2,3,4-TCDD and ^{13}C-1,2,3,7,8,9-HxCDD ISs must be within ±15 seconds of the RTs obtained during the ICAL. The RRTs of the PCDDs/PCDFs and labeled compounds must be within the limits given in Table 2 of the method.

The evaluation of RTs and subsequent qualification of sample data requires professional judgment. If RRT criteria have not been met but absolute RTs between the CCV and the ICAL and between the CCV and the sample meet criteria, qualification of data may not be necessary. If RRT criteria and absolute RT criteria are not met, this may be an indication of instrument instability warranting qualification of sample data.

Evaluation	Action
If the ion abundance ratio criteria were not met for any compound,	qualify all associated detects as "J."
If the measured concentration criteria were not met for any compound at the beginning of a 12-hour period and…	
the measured concentration is > the upper acceptance limit,	qualify all associated detects as "J+."
the measured concentration is < the lower acceptance limit,	qualify all associated detects as "J-" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."

Sample Preparation

Criteria: The cleanup standard $^{37}Cl_4$-2,3,7,8-TCDD shall be added to all extracts prior to cleanup to measure the efficiency of the cleanup process. The recovery of the cleanup standard shall be within the limits set in Table 7 of the method.

Evaluation	Action
If the cleanup standard was not added to a sample, MB, or QC sample extract,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If the recovery of the cleanup standard is > the upper acceptance limit,	qualify all associated detects as "J."
If the recovery of the cleanup standard is < the lower acceptance limit,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Sample Analysis

Criteria: For identification of any compound, the ion abundance ratios must be within the limits specified in Table 9 of the method.

The recoveries of the labeled compounds must be within the limits specified in Table 7 of the method.

The RRTs of the PCDDs/PCDFs and labeled compounds must be within the limits specified in Table 2 of the method.

Evaluation	Action
If the ion abundance ratio criteria were not met for any compound,	qualify all associated results as "R."
If the recovery of any labeled compound is > the upper acceptance limit,	qualify all detects for the corresponding unlabeled compound as "J."
If the recovery of any labeled compound is < the lower acceptance limit,	qualify all detects for the corresponding unlabeled compound as "J" and all non-detects for the corresponding unlabeled compound as "UJ".
If the RT of any compound is outside of the RT window,	qualify all associated results as "R."

Mass Spectrometer Performance Criteria

Criteria: Performance criteria are established to ensure mass resolution; identification; and, to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials. These criteria should be met in all circumstances. System performance must be evaluated at the beginning of each 12-hour period in which analysis is performed.

Evaluation	Action
If mass spectrometer performance was not evaluated at the required frequency or if method criteria were not met,	qualify all associated detects as "R" and all associated non-detects as "UJ."

GC Column Performance Mix

Criteria: The GC column performance solution is used for defining the homologous GC RT windows and to document the chromatographic resolution. Column performance must be evaluated at the beginning of each 12-hour analytical period and must meet method acceptance criteria (see Section 15.4 of the method) before sample analysis may begin.

Evaluation	Action
If GC column performance was not evaluated at the required frequency or if method criteria were not met,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Confirmation of 2,3,7,8-TCDF Detects

Criteria: The DB-5 GC column generally used for PCDD and PCDF analyses does not adequately separate 2,3,7,8-TCDF from its closest eluting isomer. If 2,3,7,8-TCDF is

detected in a sample, the result must be confirmed on a second column capable of separating 2,3,7,8-TCDF from all other TCDF homologues (as proven by successful analysis of the GC column performance column mix with <25% valley between 2,3,7,8-TCDF and its closest eluting isomer).

Evaluation	Action
If 2,3,7,8-TCDF is detected in a sample and the result is not confirmed on a second column with successful analysis of the GC column performance mix,	qualify all associated detects as "NJ."

4.3 Procedure for GC and HPLC Validation

The requirements covered within this section are applicable to all GC and HPLC analytical techniques, including SW-846 Methods 8081B, 8082A, and 8330B.

4.3.1 Calibration

Initial Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the TAL. Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing a linear curve.

When methods require confirmation of target analytes on a second, fully-calibrated column, the calibrations of both columns must be assessed.

The laboratory may establish a calibration curve using either the linear regression (linear curve) approach or the calibration factor (CF) approach. If both approaches are used to quantify and report target analytes within the same data package, calibration is to be assessed on an analyte-by-analyte basis.

Criteria: GC and HPLC instrument calibration shall be performed using a minimum of five calibration standards unless otherwise specified by the method. If calibration curves are used, five standards are required for a linear (first-order) calibration model, six standards are required for a quadratic (second-order) model, and seven standards are required for a third-order polynomial. Higher-order curves should not normally be used. If the laboratory uses a higher order equation to establish a calibration curve, it should be evaluated for appropriate application.

ISs shall not be used for quantitation.

Evaluation	Action
If an insufficient number of calibration standards were used,	qualify all associated detects as "J" and all associated non-detects as "UJ."

CFs

Criteria: In the absence of or in addition to, method-specific calibration acceptance criteria, the following general calibration acceptance criteria should be applied.

The %RSD for the CFs obtained from the five ICAL standards must be ≤20%.

Evaluation	Action
If any target compound has a %RSD:	
>20% but ≤40%,	qualify all associated detects as "J" and, all associated non-detects as "UJ" if any other calibration criteria have been exceeded for that compound.
>40% but ≤60%,	qualify all associated detects as "J" and all associated non-detects as "UJ."
> 60%,	qualify all associated detects as "J" and all associated non-detects as "R."

Linear Curves

Criteria: The r^2 of the ICAL curve shall be ≥0.99. The absolute value of the intercept shall be ≤3X the MDL.

Note: The sample results may be reported with non-detects at the MDL or at the PQL value. See below for appropriate evaluation.

Evaluation	Action
If any target compound has a r^2:	
<0.99 but ≥0.90,	qualify all associated detects as "J" and, all associated non-detects as "UJ" if any other calibration criteria have been exceeded for that compound.
<0.90 but ≥0.80,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Evaluation (concluded)	**Action (concluded)**
<0.80,	qualify all associated detects as "J" and all associated non-detects as "R."
When results are reported at the MDL: If the intercept for any target compound is negative with an absolute value… > the MDL but ≤3X the MDL, >3X the MDL,	 qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ." qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "R."
When results are reported at the PQL: If the intercept for any target compound is negative with an absolute value... > the MDL but ≤2X the PQL, >2X the PQL,	 qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ." qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "R."
If the intercept for any compound is positive and > the MDL,	qualify all associated detects <3X the intercept as "J+."

4.3.2 Calibration Verification

Compliance requirements for satisfactory initial and continuing instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the TAL. The ICV independently verifies the calibration, and the CCV establishes the relative CFs on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

Criteria: An ICV must be run immediately following an ICAL. The ICV standard analysis results are not required to be reported in the data package unless the samples in the SDG were analyzed after the ICAL standards but before a CCV

standard analysis was performed. In this case, the ICV %D is assessed according to the calibration verification criteria described below for the associated samples. If a CCV is analyzed prior to samples and ICV data are also reported in the package, both the ICV %D and the appropriate CCV %D are to be assessed as described below. If both ICV %D and CCV %D infractions occur, the worst infraction should be evaluated for result qualification.

A CCV must be run:

(1) at the beginning of each analytical run,

(2) at least once every 20 samples (preferably every 10), and

(3) at the end of each analytical run.

The laboratory is allowed to perform corrective action and reanalyze the CCV once after a failure. If multiple CCVs were analyzed (more than two) to obtain a passing CCV, then the calibration was not verified and the calibration verification frequency was not met. This is applicable to both CFs and linear curves. The evaluation of CCV data applies to all CCVs that bracket samples of interest.

A closing CCV is not required for toxaphene or chlordane if these compounds are non-detect in all samples.

Evaluation	**Action**
If the ICV/CCV standards were not analyzed at the proper frequency, or if either a required ICV or CCV was not analyzed, or if all target compounds were not present in any ICV or CCV standard,	qualify all associated detects as “J” and all associated non-detects as “UJ.”
If all required ICVs and CCVs were not analyzed,	qualify all associated detects as “J” and all associated non-detects as “R.”

CFs

Criteria: The %D (see Section 6.3) between the ICV and/or the daily or continuing calibration standard CFs and the average CFs obtained from the ICAL must be ≤15%.

Evaluation	**Action**
If the %D was reported with the wrong sign (e.g., +%D for negative bias),	document the occurrence in the data validation report and assess any infractions using the correct sign.

Evaluation (concluded)	Action (concluded)
If the %D between ICV and/or CCV CF and the average CF obtained from the ICAL is…	
>15% and positive (high bias),	qualify all associated detects as "J+."
>15% but ≤40% and negative (low bias),	qualify all associated detects as "J-." May qualify all associated non-detects as "UJ" if any other calibration criteria have been exceeded for that compound.
>40% but ≤60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
>60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "R."

Linear Curves

Criteria: The %D (see Section 6.3) between the daily or continuing calibration standard concentrations and their true values must be ≤15%.

The %D shall be calculated according to the formula in Section 6.3.

Evaluation	Action
If the %D was reported with the wrong sign (e.g., +%D for negative bias),	document the occurrence in the data validation report and assess any infractions using the correct sign.
If the %D between a measured ICV and/or CCV concentration and its true value is…	
>15% and positive (high bias),	qualify all associated detects as "J+."
>15% but ≤40% and negative (low bias),	qualify all associated detects as "J-." May qualify all associated non-detects for that compound as "UJ" if any other calibration criteria have been exceeded for that compound.
>40% but ≤60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
>60% and negative,	qualify non-detects for that compound as "R."

4.3.3 Blanks

The purpose of laboratory (or field) blank analysis is to determine the essence and magnitude of contamination resulting from laboratory (or field) activities.

The criteria for evaluation of blanks apply to any blank associated with the samples and include MBs, and, if submitted, EBs, and FBs. Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. For purposes of evaluating multiple blanks, each preparation batch may be considered an independent event in evaluating MBs, and each sampling event may be considered an independent event for evaluating FBs and EBs.

The result of any compound detected in the sample, which was also detected in any associated blank, must be qualified when the sample concentration is <5X the blank concentration

Criteria: The concentration of each target analyte found in the blank must be < the associated MDL. The sample results must not be corrected by subtracting any blank value. If QC problems exist with any blank, all data associated with the case must be carefully evaluated to determine whether there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

Evaluation	Action
If a compound found in a blank is also found a sample,	qualify the sample result for that compound in accordance with the scenarios given below.
If gross contamination exists,	qualify results for all compounds affected as “R” due to interference.
If inordinate numbers of other target compounds are found at low levels in the blank(s),	discuss the presence of these compounds in the data validation report as it may be indicative of a problem at the laboratory.

The following are examples of application of the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Scenario 1

If the sample result is > the PQL but is <5X the blank result.

Qualification

Blank Result	7
PQL	5
Sample Result	30
Qualified Sample Result	30U

Qualify sample result <35 (or 5 X 7) as non-detect ("U") at the reported value.

Scenario 2

If the sample result is < the PQL and is also <5X the blank result.

Qualification

Blank Result	6
PQL	5
Sample Result	4J
Qualified Sample Result	5U

Qualify sample result <30 (or 5 X 6) as non-detect ("U") at the PQL.

Note: Data are not reported as 4U, as this would be reported as a DL below the PQL.

The PQL may not be reported and it may not be possible to determine the PQL from the data. In these cases, qualify the contaminated sample result as "U" at 5X the blank concentration. If an MDL is reported, the PQL may be 5X the MDL.

Scenario 3

If the sample result is > the PQL and is also >5X the blank result.

Qualification

Blank Result	10
PQL	5
Sample Result	60
Unqualified Sample Result	60

Sample result exceeded the adjusted blank result of 50 (or 5 X 10). Thus, this sample result is not qualified.

4.3.4 Surrogate Recovery

Laboratory performance for individual samples is established by means of surrogate spikes. All samples are spiked with surrogate compounds prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interference and high concentrations of analytes. Because the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific surrogate results is frequently subjective and demands analytical experience and professional judgment.

Criteria: Sample and blank surrogate recoveries must be within limits specified by the laboratory. Surrogate compound recoveries shall be calculated using the procedure described in SW-846 Method 8000C. Reported recoveries shall be accompanied by the applicable acceptance limits.

Note: Results from spiked or replicate QC samples that have surrogate %Rs <10% cannot be used to qualify sample results. Samples should be qualified for lack of accuracy and/or precision data, as applicable, if specified by the program.

Evaluation	**Action**
If surrogate recovery acceptance criteria are not reported in the packages,	request amended data from the laboratory.
If, based on professional judgment, the laboratory's internal acceptance criteria are excessively wide or biased,	notify the program manager.
If an initial dilution was performed on any sample and at least one surrogate has %R < the lower acceptance limit but ≥10%, or all surrogates have <10 %R and the results for one or more compounds were ≥ the PQL,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
If an initial dilution was performed on any sample, all surrogate %Rs are <10%, and all results are < the PQL,	qualify all associated sample results as "R."
If there are two or more analyses for a particular fraction at the same dilution,	determine which analysis contains the best data to report using the considerations below, qualify all data from the rejected analysis as "R," and document the reason for rejecting data from one analysis in the data validation report.

Evaluation (concluded)	**Action (concluded)**
	Considerations should include: 1. surrogate recovery (marginal vs. gross deviation); 2. holding times; and 3. comparison of the values of the TALs reported in each fraction.
For surrogate recoveries out of specification, the following approaches are suggested based on a review of all data from the case, especially considering the apparent complexity of the sample matrix. If any surrogate %R is out of specification low, If a surrogate %R is out of specification high,	 qualify all associated detects as "J-" and all associated non-detects as "UJ" if the recovery is ≥10% and as "R" if the recovery is <10%. qualify all associated detects as "J+."

Criteria: In the case of a blank analysis with surrogates out of specification, special consideration must be given to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process.

If one or more samples in the batch show acceptable surrogate recoveries, the blank problem may be considered to be an isolated occurrence. However, even if this judgment allows some use of the affected data, analytical problems remain that must be corrected by the laboratory.

Evaluation	**Action**
If surrogate recovery in the blank does not meet acceptance criteria,	all detects < the PQL in all samples associated with the blank may be qualified as "J" and all non-detects in all samples associated with the blank may be qualified as "UJ."

4.3.5 MS/MSD

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

Criteria: The MS/MSD data shall not be used to evaluate field sample results unless the MS/MSD sample was from the same client and of similar matrix.

An MS and MSD sample shall be analyzed at a frequency of once per data package, once per 20 samples of similar matrix, or once per sample matrix, whichever is more frequent. The MS must have recoveries calculated for all single-component target compounds. The presence of multi-component target compounds in the spiking solution is recommended but not required.

The laboratory shall not use FBs or EBs to satisfy this requirement if the laboratory can identify these blanks.

Unless otherwise stated in the specific method, the MS and MSD accuracy and precision acceptance criteria shall be those calculated by the laboratory using the procedure given in SW-846 Method 8000C. If the acceptance criteria are not given, recovery limits 70% to 130% and ±30% RPD should be used as the criteria. For solid and waste samples, it may be appropriate to accept up to a 40% RPD, based on the professional judgment.

The MS %Rs must be within the limits, unless the sample concentration is >4X the spike concentration (see Section 4.1.20).

The MS and MSD analyses must meet all sample analysis acceptance criteria. The MS and MSD results may be used in conjunction with other QC results to determine the need for qualification of the data. An effort to determine to what extent the results of the MS/MSD affect the associated data should first be made. This determination should be made considering the MS/MSD sample matrix, the surrogate recoveries, and the LCS results.

Professional judgment should be used to determine if MS/MSD failure warrants qualification of only the results for the failed compounds, or if results for all the compounds associated with the failed MS compound are affected. Generally, unless evidence exists to warrant qualification of other compounds, only the compounds in the MS spiking mixture shall be qualified.

For programs that require application of one final qualifier to sample results, if a recovery (accuracy) infraction is identified in one or both of the MS samples along with an RPD (precision) infraction between the MS and MSD, the sample is qualified for the accuracy infraction. For example, if a compound has a low MS recovery and the RPD is not within criteria, the data are qualified as “J-.”

Evaluation	Action
If the program requires MS/MSD analysis for all matrices and all target compounds	

Evaluation (concluded)	**Action (concluded)**
and the MS/MSD sample was from another client or of a dissimilar matrix; the frequency of the MS/MSD did not meet specified criteria; no MS/MSD was analyzed or an FB- or EB was used for MS/MSD analysis,	qualify all detects as "J" and all non-detects as "UJ."
If no other measure of precision(i.e., LCSD or replicate) is available,	qualify all detects as "J" and all non-detects as "UJ."
If results are reported for single-component target compounds that are not in the MS,	all associated detects may be qualified as "J" and all associated non-detects may be qualified as "UJ" based on professional judgment
If any multi-component target compound is missing from the MS,	note the discrepancy in the data validation report.
If the surrogate and LCS recoveries are within the required acceptance criteria and… either MS or MSD %R for any target compound is > the upper acceptance limit, either MS or MSD %R for any target compound is < the lower acceptance limit,	 qualify all associated detects as "J+." qualify all associated detects as "J-" and all associated non-detects as "UJ" if the recovery is ≥10% and as "R" if the recovery is <10%.
If the RPD for any target compound does not meet the acceptance criteria or %Rs fail both high and low,	qualify all associated detects f as "J" and all associated non-detects as "UJ."

4.3.6 Replicate

Replicate analyses are indicators of laboratory precision based on each sample matrix. If a replicate was performed instead of an MSD, the following criteria are applied. If insufficient sample was submitted to analyze an MS/MSD or replicate, the laboratory may run an LCS/LCSD to measure precision. LCSD precision will be assessed as described in Section 4.3.5.

Criteria: Replicate samples shall be analyzed at a frequency of once per data package, once per 20 samples of similar matrix, or once per sample matrix, whichever is more frequent. All sample acceptance criteria must be met in the replicate analysis.

Samples identified as FBs or EBs shall not be used for replicate sample analysis.

Unless otherwise stated in the specific method, the replicate precision acceptance criteria shall be those calculated by the laboratory using the procedure given in SW-846 Method 8000C. When no laboratory-derived control limits are reported, a control limit of 30% for the RPD shall be used for sample values >5X the PQL. For solid and waste samples, it may be appropriate to accept up to a 40% RPD, based on the professional judgment.

A control limit of ± PQL shall be used for sample values <5X the PQL, including the case when only one of the replicate sample values is <5X the PQL.

No precision criteria apply when both replicate sample values are < the PQL.

Evaluation	**Action**
If no replicate sample, no MSD, and no LCS/LCSD was analyzed for each matrix or for each data package,	qualify all associated detects of the same matrix as "J" and all associated non-detects as "UJ."
If an FB or EB was used for the replicate analysis and no MSD or LCSD was run,	qualify all associated detects of the same matrix as "J" and all associated non-detects as "UJ."
If the original result and replicate result are both >5X the PQL, and the RPD falls outside of appropriate control limits,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."
If the original and/or replicate result is <5X the PQL (including non-detects) and the difference between the original result and replicate result is > the PQL,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."

4.3.7 LCS

Data for LCSs are generated to provide information on the accuracy of the analytical method and on laboratory performance, including sample preparation.

Criteria: An LCS should be analyzed for all methods at a frequency of once per data package, once per matrix, or once per 20 analytical samples, whichever is most frequent.

The LCS must have recovery calculated for all single-component compounds or at least one multi-component compound, if applicable. For very large analyte lists or for known poor performers, the laboratory may have received an exemption for one or more analytes. Analytes with exemptions will be identified in the case narrative.

The LCS must meet all sample acceptance criteria. If surrogate acceptance criteria are not met in the LCS analysis, the LCS must be reanalyzed. The LCS

should meet all method-specific LCS requirements and acceptance criteria. If the recovery acceptance criteria are not reported, the criteria in Appendix F or 70% to 130% should be used as the criteria.

If the laboratory analyzed an LCS/LCSD as a measure of precision, both the LCS and LCSD must meet the acceptance criteria.

General laboratory precision and accuracy can be evaluated using the LCS acceptance criteria and the interlaboratory comparison data given in Appendix F. Individual LCS recoveries may be evaluated against the criteria in Appendix F if the laboratory's criteria are significantly different from those in the tables.

Evaluation	**Action**
If, based on professional judgment, the laboratory's internal acceptance criteria are excessively wide or acceptable recoveries are significantly biased,	notify the program manager.
If the frequency of the LCS did not meet the specified criteria,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If results are reported for target compounds that are not in the LCS,	may qualify detects for these compounds as "J" and non-detects as "UJ" based on professional judgment. Compounds missing under an exemption may be qualified based on professional judgment.

If the LCS criteria are not met and reanalysis was not performed, then the laboratory performance and method accuracy are in question. Professional judgment should be used to determine if data should be qualified for all target compounds or just those compounds associated with the failed LCS compound. The following may be used as guidance in qualifying data.

Evaluation	**Action**
If the LCS %R is > the upper acceptance limit,	qualify all associated detects as "J+."
If the LCS %R is < the lower acceptance limit,	qualify all associated detects for that compound as "J-" and all associated non-detects as "UJ" if the %R is ≥10% and as "R" if the %R is <10%.

Evaluation (concluded)	Action (concluded)
If %Rs for more than half of the compounds in the LCS analysis are below the acceptance range,	qualify all associated detects as "J-", and all associated non-detects as "UJ" if the failures are marginally low and as "R" if %Rs are significantly below acceptance limits. **Note**: If %Rs for more than half of the compounds in the LCS analysis are below the acceptance range, the laboratory has not shown that it can actually meet program required DLs.
If %Rs for more than half of the compounds in the LCS analysis are above the acceptance range,	qualify all associated detects as "J+."
If %Rs for more than half of the compounds in the LCS analysis are outside the acceptance range, both above and below,	qualify all associated detects as "J" and all associated non-detects as "UJ."

4.3.8 TAL Compound Identification

These criteria are established to ensure that adequate chromatographic resolution and instrument sensitivity is achieved by the chromatographic system.

Criteria: The laboratory must report RT window data for each GC column used to analyze samples. The RT of the ICV (or the first CCV of the day) should fall within the RT window established by the ICAL. RTs of subsequent CCVs should fall within the RT window established by the ICV or the first CCV of the day.

Evaluation	Action
If RT windows were not reported,	request an amended report from the laboratory.
If RT windows are not available, or if an RT for a standard exceeds the associated windows,	qualify all associated detects as "NJ" and all associated non-detects as "R." Emphasize the possibility of either false negatives or false positives, as appropriate, in the data validation report.

4.3.9 Sample Carry-over

Sample carry-over may occur when a high-concentration sample is analyzed immediately prior to another field sample. Steps must be taken to avoid introduction of false positive results in the second sample analysis due to instrument contamination.

Criteria: The absence of sample carry-over must be determined and verified. If examination of the run logs indicates that any samples in the analytical run of interest required dilution, and there is no documentation of a rinse or blank analysis immediately following the original undiluted analysis then sample carry-over may be suspected in the subsequent sample.

Evaluation	Action
If any target compound found in the sample requiring dilution exceeded the high calibration standard and was also found in the following sample at a concentration <5X the PQL,	qualify the results for that compound in the second sample as "R" or "NJ", based on professional judgment.
If no data are available for the sample that required dilution and the laboratory has not documented that carry-over was evaluated, and the compound(s) was (were) also found in the following sample at concentrations <5X the PQL,	qualify the results for that compound in the second sample as "N."

4.3.10 Dilutions

Criteria: The PQLs must be adjusted to reflect all sample dilutions, concentrations, splits, clean-up activities, and dry weight factors that are not accounted for by the method.

Samples must be diluted and reanalyzed when any analytes exceed the calibration range. Data from original sample runs should be included when any sample requires dilution due to one or more compounds exceeding the calibration range.

The original undiluted results document the actual MDLs for non-detects.

Evaluation	Action
If the PQLs have not been properly adjusted,	request an amended report from the laboratory.

Evaluation (concluded)	**Action (concluded)**
If an initial dilution was required because of expected high concentrations of non-target analytes or because one or more target analyte were expected to greatly exceed the instrument working range and the laboratory was not able to analyze the undiluted sample,	note the dilution and elevated MDLs in the data validation report.
If any target compound exceeded the calibration range and…	
the original undiluted sample result was reported,	qualify all detects which exceeded the calibration range as "J."
the original undiluted sample run were not provided,	request this information from the laboratory.
the sample was diluted and reanalyzed and the diluted sample data were reported,	qualify all non-detects from the diluted analysis as "UJ."
If data from the original sample run are unavailable,	refer to Section 4.3.4 for assessment of initially diluted samples with low surrogate recovery.

Criteria: The laboratory shall strive to make dilutions in such a way that the final concentration is measured in the mid-range of the calibration curve, and that results are not reported from measurements below the lowest concentration standard.

Evaluation	**Action**
If the instrument response (reported result / dilution factor) of any detect from diluted samples is < that of the lowest concentration standard,	qualify all associated detects from the diluted analysis as "J."

Criteria: The extraction efficiency for extremely high concentrations of analytes has generally not been determined for most methods. If the analysis requires an extraction and dilutions of > 100,000:1 the efficiency of the extraction may be suspect.

Evaluation	Action
If dilutions of > 100,000:1 was required,	qualify all associated detects as "J-."

4.3.11 Quantification and Confirmation

Criteria: Detected compound results must be confirmed using a second GC/HPLC column. The laboratory shall report RPDs between the results obtained from the two GC/HPLC columns. RPDs are not evaluated if the analyte is not detected on the primary column. (see note below)

Evaluation	Action
If the results from the second column confirmation are not reported,	qualify all detects as "NJ."
If the RPD between detects for a particular analyte from two analytical columns is >40% and ≤ 75%… for PCB, pesticide, and herbicide analyses, for high explosive (HE) analysis,	 qualify the reported result as "J." report the result from the C-18 column and qualify it as "J."

An RPD between results for a particular analyte from two analytical columns that is >75% may indicate that there is a significant coelution or interference problem. As applied here, a coelution is two target analytes, or one target and one non-target analyte, that have peaks at the same RT, and an interference is a non-target analyte with a peak at a target analyte RT. That is, a coelution is a quantity that cannot be verified, and an interference is a result that is a false positive.

A general review of the actual spectra may be required to determine the best qualification. If the spectrum includes a significant number of extraneous peaks outside of the target analyte RT windows, interferences are likely on one or both of the columns. Non-symmetrical peak shape is indicative of coelution, and shifts in RTs may indicate either coelution or interference. A review of the beginning and ending CCV RTs will give the reviewer an indication of instrument stability during the analysis.

If one of the results is < the PQL and the other is much > the PQL, suspect interference or a false positive. Values around the PQL should be evaluated using both RPD and absolute differences. For example, results of 1 ug/L and 5 ug/L have an RPD of 133% but would not be significantly different from each other for analyses with a PQL of 5 ug/L. An attempt should be made to determine if the peak is primarily due to interference or if the peak has a significant contribution from the target analyte.

Note: It is not uncommon to find MDLs for GC/HPLC methods as determined using 40 CFR 136 to be artificially low, which may result in false positives due to random instrument noise for concentrations below the PQL.

In general, rejection of data with results much > the PQL will require additional supporting analytical information such as GC/MS or diode array spectral matching (see Appendix G).

Evaluation	**Action**
If the RPD is >75% and…	
one result is <5X the PQL and the other result is > the PQL and >10X the first result,	qualify the reported result as "R."
both results are <5X the PQL…	
for PCB, pesticide, and herbicide analyses,	qualify the reported result as "NJ."
for HE analysis,	report the result from the C-18 column and qualify it as "NJ."
both results are much > the PQL, one or both peaks may have contribution due to coelution, and…	
it is apparent that the peak is primarily due to the target analyte,	qualify the reported result as "J+."
it is not apparent that the peak is primarily due to the target analyte,	qualify the reported result as "NJ+."
If rejecting data where both results are much > the PQL,	include a complete description of the justification and supporting data used in the data validation report.

In waste-type samples, the separation techniques may not completely isolate the analytes of concern from other compounds and the spectra may contain multiple extraneous peaks. The more peaks there are in the spectra, the more likely it is that false positives will be reported.

Evaluation	**Action**
If a large number of unidentified peaks are seen in the spectra or if several additional peaks are located near a reported analyte RT in both spectra,	results may be qualified as "N" using professional judgment.

Criteria: Although confirmation is not required for non-detects it is a common laboratory practice to use a dual column system and perform the confirmation analysis on all samples. Occasionally, there may be QC failures that occur on one of the columns that are acceptable on the other column. Laboratories may choose to report the analytes with acceptable results from one column and the remaining analytes from the other column. The following guidelines should be used when this occurs.

This practice may only be used for reporting non-detects from both columns.

All QC elements must be reported for both columns.

This can only be used when no primary column is specified, such as in SW-846 Method 8082A. When a primary column is specified, such as in SW-846 Method 8330B, all QC for the primary column must be acceptable.

The QC must be completely acceptable for each analyte on one or the other column. That is, the laboratory cannot use an acceptable LCS for an analyte on one column and an acceptable CCV for that analyte on the other column to justify acceptable performance.

Evaluation	Action
If both results are reported and qualification is required,	use the results from the column with the best performance.

4.3.12 Method-specific Analytical Requirements–Organic GC and HPLC

The additional analytical requirements given below are organized by SW-846 method. These requirements should be checked if the level of deliverable (level III or level IV) allows.

4.3.12.1 Method 8081B, Organochlorine Pesticide by GC

If discussion of water sample clean-up procedures was not included in the data package, it can be assumed that clean-up was not necessary and no discussion is required in the data validation report. For soil analysis, Florisil clean-up is required for all sample extracts.

Criteria: The laboratory must include a discussion of any clean-up procedures performed on the samples.

An instrument blank consisting of clean solvent containing only the surrogate compounds shall be analyzed at the beginning and end of each analytical run, and once every 20 analytical samples.

Evaluation	Action
If discussion of sample clean-up procedures is missing or incorrect,	notify the laboratory and note the discrepancy in the data validation report.
If clean-up procedures were documented in the data package,	discuss the clean-up procedures used in the data validation report.
If no instrument blank was run, or if frequency criteria were not met,	may qualify detects <5X the MDL as "J" based on professional judgment.

Criteria: The total % breakdown for both dichlorodiphenyltrichloroethane (DDT) and endrin must each be ≤15%.

Evaluation	Action
If DDT breakdown is > 15%,	beginning with the samples following the last *in-control* standard, qualify all detects for DDT as "J" and all detects for dichlorodiphenyldichloroethane (DDD) and dichlorodiphenyldichloroethylene (DDE) as "NJ."
If DDT breakdown is >15% and DDT was not detected in any sample analyzed after the last in-control standard but DDD and DDE were detected in any of those samples,	qualify the result for DDT in the sample with DDD and DDE detects as "R."
If endrin breakdown is >15%,	beginning with the samples following the last *in-control* standard, qualify all detects for endrin as "J" and detects for endrin ketone and endrin aldehyde as "NJ."
If endrin breakdown is >15% and endrin was not detected in any sample analyzed after the last in-control standard, but endrin aldehyde and endrin ketone were detected in any of those samples,	qualify the result for endrin in the sample with the endrin aldehyde and endrin ketone detects as "R."

Note: A closing CCV is not required for toxaphene or chlordane if these compounds are non-detect in all samples.

4.3.12.2 Method 8082A, PCB Aroclors by GC

Criteria: PCB analysis shall be performed according to the requirements listed in SW-846 Method 8082A.

The laboratory must include a discussion of any clean-up procedures performed on the samples. If discussion of sample clean-up procedures was not included in the data package, it can be assumed that clean-up was not necessary and no discussion is required in the data validation report. The laboratory case narrative shall include a thorough discussion of any problems encountered regarding target compound recognition and/or quantitation and especially addressing suspected environmental degradation of compounds. Reported results shall be justified with such discussion and supporting documentation.

PCBs reported as total PCBs or as individual congeners are qualified in accordance with the Section 4.2.14.6.

Evaluation	Action
If clean-up procedures were documented in the data package,	discuss the clean-up procedures used in the data validation report.
If the discussion does not appear to justify the results reported by the laboratory,	notify the laboratory; more supporting documentation may be required from the laboratory.
If the laboratory identifies any aroclors as degraded,	qualify all associated detects as “J.”

4.3.12.3 Method 8151A, Chlorinated Herbicides by GC

Criteria: Chlorinated herbicide analysis shall be performed according to the requirements listed in the SW-846 Method 8151A.

The LCS shall contain each of the specified target chlorinated herbicides at concentrations near the midpoint of the calibration range.

Evaluation	Action
If results are reported for target compounds that are not in the LCS,	may qualify all associated detects as “J” based on professional judgment.
If LCS analytes are not at concentrations near the midpoint of the calibration range,	note the finding in the data validation report and notify the laboratory.

4.3.12.4 Confirmation of Polyaromatic Hydrocarbon (PAH) by Method 8310

The primary analysis should be done by HPLC on a C18 column using a diode array detector. Confirmation is done qualitatively using spectral matching and/or quantitatively using a fluorescence detector. This method presupposes a high expectation of detecting the compounds. When used as a screening tool both confirmation methods should be employed. If a co-eluting compound is present that is detected by both the diode array detector and the fluorescence detector, the primary method of determining if interference is present is the spectral match from the diode array detector.

An effort to determine if the peak is primarily from the target compound or due to interference should be made. This is determined by comparison of the sample diode array spectra to the reference spectra in accordance with Appendix G.

Evaluation	**Action**
If the diode array spectra were used for confirmation and no diode array spectra were included in the data package,	qualify the result as "NJ."
If the sample absorption spectra does not match the standard absorption spectra or the percent difference spectra does not exhibit a relatively straight line,	qualify the result as "R."
If the analyte was misidentified by the laboratory,	request an amended report from the laboratory.
If identification of the analyte was hampered by interferences such that it is not certain that a positive identification could be made or that the quantification may be biased high,	qualify all associated results as "N" or "NJ" based on professional judgment or request additional data from the laboratory.

The second evaluation compares the calculated values from the two detectors when a two-detector system is used. When one of the results is < the PQL and the other is much > the PQL (i.e., near or above the mid-point in the calibration curve), suspect interference or a false positive. Values around the PQL should be evaluated using both the RPDs and absolute differences. For example, results of 1 ug/L and 5 ug/L have an RPD of 133% but would not be significantly different from each other for analyses with a PQL of 5 ug/L.

Evaluation	**Action**
If results from the second column confirmation were not reported,	qualify all detects as "NJ."
If the RPD between detects for a particular analyte from two analytical columns is >40% and ≤75%,	report the result from the diode array detector and qualify it as "J."

Evaluation (concluded)	Action (concluded)
If the RPD is > 75% and…	**Note**: If the RPD is >75%, one or both peaks may be due to coelution.
one result is <5X the PQL and the other result is > the first result,	qualify the result as "R."
both results are <5X the PQL,	qualify the result as "NJ."
both results are >5X the PQL and it appears that the peak is primarily due to the target analyte (spectral match),	qualify the result as "J+."
both results are >5X the PQL and it is not apparent that the peak is primarily due to the target analyte,	qualify the result as "NJ+."
If rejecting data where both results are much > the PQL,	include a complete description of the justification for the rejection and supporting data used in the data validation report. **Note**: In general, rejection of data with results >5X the PQL will require additional supporting analytical information such as GC/MS spectral matching.

4.3.12.5 Method 8015C or 8015D, Non-halogenated Organics Using GC/Flame Ionization Detector (Gasoline Range Organics/Diesel Range Organics)

Confirmation on a second column is not typically required for gasoline range organics (GRO) and diesel range organics (DRO) reported by this method.

GRO and DRO results represent all peaks detected over a designated RT range on the chromatogram. The RT assessment is performed as described in Section 4.3.8 for all reported RT markers.

Evaluation	Action
If RT windows are exceeded,	qualify all associated detects as "J" and all associated non-detects as "UJ."

4.4 Procedure for Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) Validation

The requirements addressed within this section are applicable to all LC/MS/MS analytical techniques. LC/MS/MS is a highly selective analysis that utilizes four means of compound discrimination: chromatographic separation, negative ion generations (where applicable), mass selection, and daughter fragmentation. It is theoretically possible that two different compounds could have the same RT and generate the same ion, but it is highly unlikely that these two compounds would fragment to the same daughter ion.

4.4.1 Instrument Calibration for LC/MS/MS

Initial Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the TAL. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run and of producing a linear curve. In the absence of or in addition to, method-specific calibration acceptance criteria, the following general calibration acceptance criteria should be applied.

If an IS is used to calculate analytical results, the slope or RF values are evaluated as directed below. If the analysis does not use an IS to quantitate analytical results, the value of the slope or CF is not evaluated.

The laboratory may establish a calibration curve using either the linear regression (linear curve) approach or the RF approach. If both approaches are used to quantify and report target analytes within the same data package, calibration is to be assessed on an analyte-by-analyte basis.

Criteria: LC/MS/MS instrument calibration shall be performed using a minimum of five calibration standards. The lowest point of the curve must be at or below the PQL.

If calibration curves are used, five standards are required for a linear (first-order) calibration model, six standards are required for quadratic (second-order) model, and seven standards are required for third-order polynomial. Higher order curves should not normally be used. If the laboratory uses a higher-order equation to establish a calibration curve, it should be evaluated for appropriate application.

Daily instrument calibration is required for perchlorate analysis.

Evaluation	Action
If an insufficient number of calibration standards were used, the PQLs were incorrect or all points were not analyzed within a 24-hour period:	qualify all associated detects as “J” and all associated non-detects as “UJ.”

Evaluation (concluded)	Action (concluded)
If the instrument for perchlorate analysis was not calibrated daily,	qualify all associated detects as "J" and all associated non-detects as "UJ."

RFs

Criteria: The %RSD for the RFs obtained from the five ICAL standards must be ≤20%.

Evaluation	Action
For analyses using an IS for analyte quantitation, if the average RF for any target analyte is < the specified minimum RF, or <0.05 if no minimum is specified,	qualify all associated detects as "J" and all associated non-detects as "UJ" if the RF is ≥0.01 and as "R" if the RF is <0.01.
If any target compound has a %RSD…	
>20% but ≤40%,	qualify all associated detects as "J" and, may qualify all associated non-detects as "UJ" if any other calibration criteria have been exceeded for that compound.
>40% but ≤60%,	qualify all associated detects as "J" and all associated non-detects as "UJ."
> 60%,	qualify all associated detects as "J" and all associated non-detects as "R."

Linear Curves

Criteria: The r^2 of the ICAL curve shall be ≥0.99 and have a slope ≥0.05 for each compound. The absolute value of the intercept shall be ≤3X the MDL.

Note: The sample results may be reported with non-detects at the MDL or at the PQL value. See below for appropriate evaluation.

For perchlorate, forcing the calibration curve through a zero intercept is an acceptable practice and usually results in more accurate quantitation for low level results.

Evaluation	**Action**
For analyses using an IS for analyte quantitation, if the slope for any target analyte is < the specified minimum RF, or <0.05 if no minimum RF is specified,	qualify all associated detects as "J" all associated non-detects as "UJ" if the slope is ≥0.01 and as "R" if the slope is < 0.01.
If any target compound has a r^2: <0.99 but ≥0.90, <0.90 but ≥0.80, < 0.80,	 qualify all associated detects as "J" and may qualify all associated non-detects as "UJ" if any other calibration criteria have been exceeded for that compound. qualify all associated detects as "J" and all associated non-detects as "UJ." qualify all associated detects as "J" and all associated non-detects as "R."
If the intercept for any target analyte is positive and > the MDL,	qualify all associated detects <3X the intercept as "J+."
When results are reported at the MDL: If the intercept for any target compound is negative with an absolute value... > the MDL but ≤3X the MDL, >3X the MDL,	 qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ." qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "R."
When results are reported at the PQL: If the intercept for any target compound is negative with an absolute value... > the MDL but ≤2X the PQL,	 qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ."

Evaluation (concluded)	Action (concluded)
>2X the PQL,	qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "R."

4.4.2 Calibration Verification

Compliance requirements for satisfactory initial and continuing instrument calibration verification are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for target compounds. The ICV independently verifies the calibration, and CCV establishes the 12-hour relative RFs on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

The evaluation of CCV data applies to all CCVs that bracket samples of interest.

Criteria: An ICV standard is analyzed immediately following an ICAL. For perchlorate analysis, the ICV is always evaluated for %D criteria. For HE analysis, the ICV standard analysis results are not required to be reported in the data package unless the samples in the SDG were analyzed after the ICAL but before a CCV standard analysis was performed. In this case, the ICV %D is assessed according to the calibration verification criteria described below for the associated samples. If a CCV is analyzed prior to samples and ICV data are also reported in the package, both the ICV %D and the appropriate CCV %D are to be assessed as described below. If both ICV %D and CCV %D infractions occur, the worst infraction should be evaluated for result qualification.

A CCV standard must be analyzed:

1) at the beginning of each analytical run;
2) at least once every 10 samples; and
3) and at the end of each analytical run.

If multiple CCVs were analyzed to obtain a passing CCV, the calibration is not verified and the calibration frequency is not met.

Evaluation	Action
If the ICV/CCV standards were not analyzed at the proper frequency, or if either a required ICV or CCV was not analyzed, or if all target compounds were not present in any ICV or CCV standard,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If all required ICVs and CCVs were not analyzed,	qualify all associated detects as "J" and all associated non-detects as "R."

RFs

Criteria: The %D between the ICV and/or CCV RFs and the average RF obtained from the ICAL shall be calculated according to the formula in Section 6.3 and must be ≤20% for HE and ≤15% for perchlorate. The evaluation of CCV data applies to all CCVs that bracket samples of interest.

Evaluation	Action
If the %D was reported with the wrong sign (e.g., +%D for a negative bias),	document the occurrence in the data validation report and assess any infractions using the correct sign.
If the %D between an ICAL RF or CF and an ICV or CCV RF or CF for any target analyte is...	
>20% for HE or >15% for perchlorate and positive (high bias),	qualify all associated detects as "J+."
>20% for HE or >15% for perchlorate but ≤40% and negative (low bias),	qualify all associated detects as "J-" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>40% but ≤60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
>60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "R."

Linear Curves

Criteria: The %D between the ICV or CCV standard concentrations and their true values shall be calculated according to the formula in Section 6.3 and must be ≤20% for HE and ≤15% for perchlorate. The evaluation of CCV data applies to all CCVs that bracket samples of interest.

Evaluation	Action
If the %D was reported with the wrong sign (e.g., +%D for negative bias),	document the occurrence in the data validation report and assess any infractions using the correct sign.

Evaluation (concluded)	Action (concluded)
If the %D between a measured ICV and/or CCV concentration and its true value for any analyte is...	
>20% for HE or >15% for perchlorate and positive (high bias),	qualify all associated detects as "J+."
>20% for HE or >15% for perchlorate but ≤40% and negative (low bias),	qualify all associated detects as "J-" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."
>40% but ≤60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
>60% and is negative,	qualify all associated detects as "J-" and all associated non-detects as "R."

4.4.3 RLV

A RLV standard (i.e., RLV for ICP-AES, ICP-MS, and LC/MS/MS methods [CRI]), of the same origin as the calibration standard must be analyzed at the beginning and end of each perchlorate analysis run and at the beginning only if each HE analytical run as a measure of accuracy near the PQL. This analysis may be referred to as a PS. Analysis of a CRI is required for both HE and perchlorate methods. CRI standard concentrations are at 2X the MDLs for perchlorate analysis, and at no more than 2X the PQL for HE analysis.

The laboratory may run more than the required CRIs in a batch. In this case, the bracketing CRIs for perchlorate and the CRI immediately preceding the samples for HE will be used for the CRI evaluation.

Criteria: The advisory recovery acceptance criteria for perchlorate analysis are 70% to 130%. For HE analysis, recoveries must be within limits specified by the laboratory. If recovery acceptance criteria are not reported, the recovery acceptance range shall be 70% to 130%.

Evaluation	Action
If frequency criteria are not met,	qualify all detects <5X the PQL as "J" and all non-detects as "UJ."
If the %R is >130%,	qualify all associated detects <5X the PQL as "J+."

Evaluation (concluded)	Action (concluded)
If the %R is <70% but ≥30%,	qualify all associated detects <5X the PQL as "J-" and all associated non-detects as "UJ."
If the %R is <30%,	qualify all associated detects <5X the PQL as "J-" and all associated non-detects as "R."

4.4.4 Blanks

For perchlorate analysis, refer to Section 4.5.3 for assessment of blanks.

The following applies for HE analysis.

The preparation batch consists of a group of no more than 20 samples of the same matrix processed on the same day. All samples in a batch must be initiated on the same day. Each batch must contain a MB.

An initial calibration blank (ICB) must be analyzed to verify the baseline immediately following calibration and prior to analytical sample analysis. A continuing calibration blank (CCB) must be analyzed after each CCV and at the end of every analytical sequence in order to bracket all sample analyses. All CCBs that bracket samples of interest shall be reported and assessed. If a bracket has an ICB and no CCB, then the ICB should be treated as a CCB for validation purposes.

The purpose of laboratory (or field) blank analysis is to determine the essence and magnitude of contamination resulting from laboratory (or field) activities.

The criteria for evaluation of blanks apply to any blank associated with the samples and include MBs and, if submitted, EBs and FBs. Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. For purposes of evaluating multiple blanks, each preparation batch may be considered an independent event in evaluating preparation blanks, and each 12-hour run sequence may be considered an independent event for evaluating FBs and EBs.

The result of any compound detected in the sample, which was also detected in any associated blank, must be qualified when the sample concentration is <5X the blank concentration.

Criteria: The concentration of each target analyte found in the blank must be < the associated MDL. The sample results must not be corrected by subtracting any blank value. If QC problems exist with any blank, all data associated with the. case must be carefully evaluated to determine whether there is an inherent

variability in the data for the case, or if the problem is an isolated occurrence not affecting other data

Evaluation	Action
If a compound found in a blank is also found in the field sample,	qualify the sample result for that compound in accordance with the scenarios given below.
If gross contamination exists,	qualify results for all compounds affected as "R" due to interference.
If inordinate numbers of other target compounds are found at low levels in the blank(s)	discuss the presence of those compounds in the data validation report as it may be indicative of a problem at the laboratory.

The following are examples of application of the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Scenario 1

Sample result is > the PQL but is <5X the blank result.

Qualification

Blank Result	7
PQL	5
Sample Result	30
Qualified Sample Result	30U

Sample results <35 (or 5 X 7) would be qualified as non-detects ("U") at the reported value.

Scenario 2

The sample result is < PQL and is also <5X the blank result.

Qualification

Blank Result	6
PQL	5
Sample Result	4J
Qualified Sample Result	5U

Qualify sample results <30 (or 5 X 6) as non-detect ("U") at the PQL.

Note: Data are not reported as 4U, as this would be reporting a DL below the PQL.

The PQL may not be reported and it may not be possible to determine the PQL from the data. In these cases, qualify the contaminated sample result as "U" at 5X the blank concentration.

Scenario 3

The sample result is > the PQL and is also >5X the blank result.

Qualification

Blank Result	10
PQL	5
Sample Result	60
Unqualified Sample Result	60

Sample result exceeded the adjusted blank result of 50 (or 5 X 10). Thus, this sample result is not qualified.

4.4.5 Surrogate Recovery – HE analysis only

Laboratory performance on individual samples is established by means of surrogate spikes. All samples are spiked with surrogate compounds prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interference and high concentrations of analytes. Because the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. The evaluation of surrogate recoveries and ISs should be performed concurrently. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

Criteria: Sample and blank surrogate recoveries must be within limits specified by the laboratory. Surrogate compound recoveries shall be calculated using the procedure described in SW-846 Method 8000C. Reported recoveries shall be accompanied by the applicable acceptance limits.

Results from spiked or replicate QC samples that have surrogate %Rs < 10% cannot be used to evaluate associated sample results. Associated samples should be qualified for lack of accuracy and/or precision data as applicable.

Evaluation	Action
If the surrogate recovery acceptance criteria were not reported in the data package,	request amended data from the laboratory.

Evaluation (concluded)	**Action (concluded)**
If, based on professional judgment, the laboratory's internal acceptance criteria are excessively wide or biased,	notify the program manager.
If an initial dilution was performed on any sample and at least one surrogate recovery is < the lower acceptance limit but ≥10%, or all surrogate recoveries are <10% and the results for one or more compounds are > the PQL,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
If an initial dilution was performed on any sample, any surrogate %R is <10%, and all results are non-detect,	qualify all sample results as "R."
If there are two or more analyses for a particular fraction at the same dilution,	determine which contains the best data to report using the considerations below, qualify all data from the rejected analysis as "R" and document the reason for rejecting data from one analysis in the data validation report. Considerations should include: 1. surrogate recovery (marginal vs. gross deviation); 2. holding times; 3. comparison of the values of the target analytes reported in each fraction; and 4. performance of ISs.
For surrogate spike recoveries out of specification, the following approaches are suggested based on a review of all data from the batch, especially considering the apparent complexity of the sample matrix.	
If the surrogate is out of specification low,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the recovery is ≥10% and as "R" if the recovery is <10%.
If the surrogate is out of specification high,	qualify all detects as "J+."

Criteria: In the case of a blank analysis with surrogate out of specification, special consideration must be given to the validity of associated sample data. The basic

concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process.

If one or more samples in the batch show acceptable surrogate recovery, r the blank problem may be considered to be an isolated occurrence. However, even if this judgment allows some use of the affected data, analytical problems remain that must be corrected by the laboratory.

Evaluation	Action
If surrogate recovery in the blank does not meet acceptance criteria,	may qualify all detects < the PQL in all samples associated with the blank as "J" and all non-detects in all samples associated with the blank as "UJ."

4.4.6 IS Performance

IS criteria ensure that instrument sensitivity and response are stable and acceptable during each analysis.

Criteria: The laboratory may use an IS to calculate the result, or it may use the IS as a RT check only (perchlorate). If the IS is used for quantification, the IS area counts must not vary by more than 70% to 130% and the RT of the IS must not vary by more than ±30 seconds from the average of those obtained from the calibration standards or from the mid-level calibration standard.

If the IS is only used as an RT check, the RRT of the IS must fall within the acceptance range of 0.98 to 1.02, and the IS recovery should be evaluated using the surrogate criteria. If recovery acceptance limits are not reported in the data package, recovery should be evaluated based on reported MS acceptance limits.

Evaluation	Action
If there are two analyses for a particular sample,	determine which contains the best data to report based on the considerations below, qualify all data from the rejected analysis as "R" and document the reason for rejecting data from one analysis in the data validation report. Considerations should include: 1. magnitude of the RT shift; 2. holding times; 3. comparison of the values of the target compounds reported in each sample; and. 4. surrogate recovery.

Evaluation (concluded)	**Action (concluded)**
If the IS was used for quantification and...	
its area count is >130% of the average of that obtained from the calibration standards,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
its area count is <70% of the average of that obtained from the calibration standards,	qualify all associated detects as "J+" and all associated non-detects as "UJ" if the area count is ≥25% and as "R" if the area count is <25%. **Note:** If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated.
its RT varies by more than ±30 seconds,	qualify all associated detects as "N" or "R" and all associated non-detects as "R."
If the IS was used as an RT check and the RRT does not fall within the acceptance range,	qualify all associated detects as "N" or "R" and all associated non-detects as "R."
If the IS was used as an RT check,	evaluate the IS area counts according to Section 4.4.5.

4.4.7 MS/MSD

Data for MS/MSD pairs are generated to determine long-term precision and accuracy of the analytical method on samples various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

Criteria: The MS/MSD data shall not be used to evaluate associated field sample results unless the MS/MSD sample was from the same client and of similar matrix.

An MS and MSD sample shall be analyzed at a frequency of once per data package, once per 20 samples of similar matrix, or once per sample matrix, whichever is more frequent.

The laboratory shall not use FBs or EBs to satisfy these requirements if the laboratory can identify these blanks.

For HE, the MS and MSD accuracy and precision acceptance criteria shall be those calculated by the laboratory using the procedure given in SW-846 Method 8000C. If the recovery acceptance criteria are not given, recovery limits of 70% to 130% and 30% RPD should be used as the criteria. For solid and waste samples, it may be appropriate to accept up to a 40% RPD, based on the professional judgment of the reviewer.

For perchlorate, the MS/MSD recovery acceptance criteria are 75% to 125% and 20% RPD. For solid and waste samples, it may be appropriate to accept up to a 30% RPD, based on the professional judgment.

The MS and MSD %R must be within the limits, unless the sample concentration is >4X the spike concentration (see Section 4.1.20).

The MS and MSD analyses must meet all sample analysis acceptance criteria. The MS and MSD may be used results in conjunction with other QC results to determine the need for qualification of the data. An effort to determine to what extent the results of the MS/MSD affect the associated data should first be made. This determination should be made considering the MS/MSD sample matrix, the surrogate and IS %Rs, and the LCS results. Professional judgment should be used to determine if MS/MSD failure warrants qualification of only the results for the failed compounds or if results for all the compounds associated with the failed MS compound and its associated IS are affected. Generally, unless evidence exists to warrant qualification of other compounds, only the compounds in the MS spiking mixture shall be qualified.

For programs that require application of one final qualifier to sample results, if a recovery (accuracy) infraction is identified in one or both of the MS samples along with an RPD (precision) infraction between the MS and MSD, the sample is qualified for the accuracy infraction. For example, if an analyte has low MS recovery and the RPD is not within criteria, the data are qualified as “J-.”

Evaluation	**Action**
If the MS/MSD sample was from another client or of a dissimilar matrix; the frequency of the MS/MSD did not meet specified criteria; no MS/MSD was analyzed; an FB or EBs was used for MS/MSD purposes.	qualify all detects as “J” and all non-detects as “UJ.”
If no other measure of precision (i.e., LCSD or replicate) is available,	qualify all detects as “J” and all non-detects as “UJ.”
If the surrogate, IS and LCS %Rs are within the required acceptance criteria and…	

Evaluation (concluded)	Action (concluded)
either %R for any target compound is > the upper acceptance limit,	qualify all associated detects as "J+."
either %R for any target compound is < the lower acceptance limit, and ≥ 10%,	qualify all associated detects as "J-" and all associated non-detects as UJ" if the recovery is ≥10% and as "R" if the recovery is <10%.
If the RPD for any target compound does not meet the acceptance criteria or %Rs fail both high and low,	qualify all associated detects as "J" and all associated non-detects as "UJ."

4.4.8 Replicate

Replicate analyses are indicators of laboratory precision based on each sample matrix. If a replicate was performed instead of a MSD, the following criteria are applied. If insufficient sample was submitted to analyze a MS/MSD or replicate, the laboratory may run an LCSD to measure precision.

Criteria: A replicate sample shall be analyzed at a frequency of once per data package, once per 20 samples of similar matrix, or once per sample matrix, whichever is more frequent. All sample acceptance criteria must be met in the replicate analysis.

Samples identified as FBs or EBs should not be used for replicate sample analysis.

For HE values ≥5X the PQL, the replicate precision acceptance criteria shall be that calculated by the laboratory using the procedure given in SW-846 Method

8000C. If the acceptance criteria are not given, an RPD of ≤30% should be used as the acceptance criteria. For solid and waste samples, it may be appropriate to accept up to a 40% RPD, based on the professional judgment.

For perchlorate, the replicate precision acceptance criteria is 20% RPD for sample values ≥5X the PQL. For solid and waste samples, it may be appropriate to accept up to a 30% RPD, based on the professional judgment.

For both HE and perchlorate analysis, a control limit of ± the PQL shall be used for sample values > the PQL but <5X the PQL, including the case when only one of the replicate sample values is > the PQL but <5X the PQL.

No precision criteria apply when both replicate sample values are < PQL.

Evaluation	Action
If a replicate sample, MSD, and LCS/LCSD were not analyzed for each matrix or for each data package,	qualify all detects of the same matrix as "J" and all non-detects of the same matrix as "UJ."

Evaluation (concluded)	Action (concluded)
If an FB or EB was used for the replicate analysis and no MSD or LCSD was run,	qualify all detects of the same matrix as "J" and all non-detects of the same matrix as "UJ."
If the original result and replicate result for any target compound are both ≥5X the PQL, and the RPD exceeds the appropriate control limit,	qualify all associated detects of the same matrix as "J" and all associated non-detects all associated as "UJ."
If the original and/or replicate result for any target compound is > the PQL but <5X the PQL (including non-detects) and the difference between the original result and replicate result is > the PQL,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."

4.4.9 LCS

Data for LCSs are generated to provide information on the accuracy of the analytical method and on laboratory performance including sample preparation.

Criteria: An LCS should be analyzed for all methods at a frequency of once per data package, once per matrix, or once per 20 analytical samples, whichever is most frequent.

The LCS must meet all sample acceptance criteria and all method-specific LCS requirements. The LCS for HE must meet laboratory-derived acceptance criteria. If surrogate and IS recovery acceptance criteria are not met for the LCS analysis, the LCS must be reanalyzed. If the recovery acceptance criteria are not reported, the criteria in Appendix F or 70% to 130% should be used as the criteria.

The recovery acceptance limits for perchlorate are 85% to 115%.

If the laboratory analyzed an LCS/LCSD as a measure of precision, both the LCS and LCSD must meet the acceptance criteria.

Evaluation	Action
If, based on professional judgment, the laboratory's internal acceptance criteria are excessively wide or acceptable recoveries are significantly biased,	notify the program manager.
If the frequency of the LCS did not meet the specified criteria,	qualify all detects as "J" and all non-detects as "UJ."

Evaluation (concluded)	**Action (concluded)**
If results are reported for target compounds that are not in the LCS,	may qualify detects for those compounds as "J" and non-detects for those compounds may be qualified as "UJ" based on professional judgment.

If the LCS criteria are not met and reanalysis was not performed, then the laboratory performance and method accuracy are in question. The following may be used as guidance in qualifying data.

Evaluation	**Action**
If the LCS %R is > the upper acceptance limit,	qualify all associated detects as "J+."
If the LCS %R is < the lower acceptance limit,	qualify all associated detects as "J-" and all associated non-detects as "UJ" if the %R is ≥ 10% and as "R" if %R is < 10%.
For HE analysis, if %Rs for more than half of the compounds in the LCS analysis are below the acceptance range,	qualify all detects as "J-" and all non-detects as "UJ" if the %Rs are marginally low and as "R" if %Rs are significantly below acceptance limits.
For HE analysis, if %Rs for more than half of the compounds in the LCS analysis are above the acceptance range,	qualify all detects as "J+."
For HE analysis, if %Rs for more than half of the compounds in the LCS analysis are outside the acceptance range, both above and below, or if an LCS/LCSD pair was analyzed and the recoveries of any target analyte are both above and below acceptance criteria,	qualify all detects as "J" and non-detects as "UJ."

4.4.10 Sample Carry-over

Sample carry-over may occur when a high-concentration sample is analyzed immediately prior to another field sample. Steps must be taken to avoid introduction of false positive results in the second sample analysis due to instrument contamination.

Criteria: The absence of sample carry-over must be determined and verified. If examination of the run logs indicates that any samples in the analytical run of interest required dilution and there is no documentation of a rinse or blank analysis immediately following the original undiluted analysis then sample carry-over may be suspected.

Evaluation	**Action**
If any compound found in the sample requiring dilution exceeds the high calibration standard and was also found in the following sample at concentration <5X the PQL,	qualify the results for that compound in the second sample as "R."
If no data are available for the sample that required dilution and the laboratory has not documented that carry-over was evaluated, and any compound was also found in the following sample at concentration <5X the PQL,	qualify the results for that compound in the second sample as "N."

4.4.11 Dilutions

Criteria: The PQLs must be adjusted to reflect all sample dilutions, concentrations, splits, clean-up activities, and dry weight factors that are not accounted for by the method.

Samples must be diluted and reanalyzed when any analyte exceeds the calibration range.

Original sample runs should be included when any sample requires dilution due to one or more compounds exceeding the calibration range.

The original undiluted results document the actual MDLs for non-detects.

Evaluation	**Action**
If the PQLs have not been properly adjusted,	request an amended report from the laboratory.
If an initial dilutions was required because of expected high concentrations of non-target analytes or because one or more target analytes were expected to greatly exceed the instrument working range and the laboratory was not be able to analyze the undiluted sample.	note the dilution and evaluated MDLs in the data validation report.

Evaluation (concluded)	Action (concluded)
If any target compound exceeds the calibration range and…	
the original undiluted sample result was reported,	qualify all detects from the undiluted analysis that exceed the calibration range as "J."
the sample is diluted and reanalyzed, and the diluted sample data were reported,	qualify all non-detects from the diluted analysis as "UJ."
the original undiluted sample data was not provided,	request this information from the laboratory.
If data from the original sample run are unavailable,	refer to Section 4.2.5 for assessment of initially diluted samples with low surrogate recovery.

Criteria: The laboratory shall strive to make dilutions in such a way that the final concentration is measured in the mid-range of the calibration curve, and that results are not reported from measurements below the lowest concentration standard.

Evaluation	Action
If the instrument response (reported result / dilution factor) from diluted sample is < that of the lowest concentration standard,	qualify all detects from the diluted analysis as "J."

4.4.12 Perchlorate Chlorine Ratios

Criteria: The natural isotopic abundances for the chlorine isotopes give a $^{35}Cl/^{37}Cl$ ratio of approximately 3.08. Laboratories must statistically derive isotope ratio acceptance criteria to be used as an additional confirmation of analyte identity.

When the laboratory does not specify acceptance criteria the mean of the ratio population shall not deviate by more than 10% from the 3.07 theoretical value, and the standard deviation shall not significantly exceed 0.2. Between the MDL and the PQL, the individual sample isotope ratio control limits shall be near the population mean ±20% (approximately 3σ). Above the PQL, the individual sample isotope ratio control limits shall be near the population mean ±15% (approximately 2σ).

When isotope ratio acceptance criteria are not met, the laboratory must provide supporting data and explanatory case narrative comments in the data package.

Evaluation	Action
If the isotope ratios were not reported,	calculate the ratio if the raw data were supplied or request an amended report from the laboratory if the raw data were not supplied.
If the isotope ratios are outside acceptance limits,	qualify detects as "NJ" or "R" based on professional judgment.
If supporting data and explanation were not provided,	request an amended report from the laboratory.

4.4.13 Perchlorate Interference Check Standard

Criteria: The laboratory shall analyze an interference check standard (ICS) from a matrix containing 500 ppm each of chloride, sulfate, carbonate, and bicarbonate in every batch. The concentration of this standard will be at the PQL. To demonstrate that perchlorate is adequately isolated and recovered under the specific conditions used, this standard should recover within ±20% of the known value.

Evaluation	Action
If frequency criteria were not met,	note the deficiency in the data validation report.
If the recovery is not within ±20% of the known value,	note the deficiency in the data validation report.

4.4.14 Method-specific Analytical Requirements – Organic LC/MS/MS

The additional analytical requirements addressed below are organized by method. These requirements should be checked if the level of deliverable allows.

4.4.14.1 Method 1694: Pharmaceuticals and Personal Care Products (PPCP) in Water, Soil, Sediment, and Biosolids by HPLC/MS/MS

Validation is to be performed as data deliverables allow.

Note: MS/MSD and replicate analyses are not required for Method 1694.

Initial Calibration

Criteria: If isotope dilution calibration is used, the %RSD must be <20%. If IS calibration is used, the %RSD must be <35%. Absolute RTs for last-eluting compounds in each of the four calibration groups must be ≥ the reference RT listed in Tables 3, 5, 7, and 9 of Method 1694.

Evaluation	Action
If the %RSD is: >20% for any compound calibrated using isotope dilution method or >35% for any compound calibrated using IS calibration, but ≤ 40%, >40% but ≤60% for either a labeled or unlabeled calibration standard, >60% for either a labeled or unlabeled calibration standard,	 qualify all associated detects as "J" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ." qualify all associated detects as "J" and all associated non-detects as "UJ." qualify all associated detects as "J" and all associated non-detects as "R."
If the RT of the last eluting compound in any of the four analysis groups is < the reference RT,	qualify all associated detects as "J" and all associated non-detects as "UJ."

Calibration Verification

Criteria: A QC Check Sample (from a second source vendor, similar to an ICV) shall be analyzed at least quarterly. The CCV %D must be ±30% of the ICAL response factor for all labeled and unlabeled compounds. The LC peaks for all native and labeled compounds must be present with a signal to noise ratio of at least 10. The retention times of the native and labeled compounds must be within 15 seconds (0.25 minutes) of the respective RTs in the most recent calibration verification standard.

Evaluation	Action
If there is no evidence in the data package of a QC Check sample (ICV) analysis,	note in the data validation report without qualification.
If the %D between an ICAL RF and an ICV or CCV RF for any labeled or unlabeled compound is... >30% and positive (high bias), >30% and negative (low bias),	 qualify all associated detects as "J+." qualify all associated detects as "J-" and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as "UJ."

Evaluation (concluded)	**Action (concluded)**
>40% but ≤60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
>60% and negative,	qualify all associated detects as "J-" and all associated non-detects as "R."

Evaluation	**Action**
If the LC peaks for all native and labeled compounds do not have a signal to noise ratio of ≥10,	qualify detects as "J_" and non-detects as "UJ."
If the LC peaks for all native and labeled compounds do not have a signal to noise ratio of ≥3,	qualify detects as "J-" and non-detects as "R."

Evaluation	**Action**
If any native or labeled compound RT has shifted more than 15 seconds (0.25 minutes) from the RT of the most recent calibration verification standard,	qualify detects as "J_" and non-detects as "R".

Labeled Compound Recovery

Criteria: Labeled Compound recovery must meet criteria given in Table 12 of the method for LCS (OPR) samples, and field samples.

Evaluation	**Action**
If the labeled compound %R is < method acceptance criteria lower acceptance limit but >2.5%,	qualify all associated detects for that sample fraction as "J+" and all non-detects for that sample fraction as "UJ."
If the labeled compound %R for is <2.5%,	qualify all results for the associated result as "R."
If the %R for any sample fortification solution compound is > the upper limit of the method acceptance criteria	qualify all detects for that sample fraction as "J-" and all non-detects for that sample fraction as "UJ."

Surrogate Recovery

Follow criteria listed in Section 4.4.5.

IS Performance

An IS performance criterion is not given in Method 1694.

Target Analyte Identification

Criteria: The signal to noise ratio for each native compound identified above the MDL in a sample, MB, or LCS must be ≥2.5, and ≥10 in associated calibration standards, except for the lowest ICAL standard, which must have a signal to noise ratio of 3. The RT of the peak for all identified compounds must be ±15 seconds (0.25 minutes) of the most recent CCV.

Evaluation	Action
If signal to noise ratio and/or RT criteria are not met for any reported compound,	notify the program manager and request an amended report.

4.5 Validation Guidelines for Confirmation by LC/MS/MS

These guidelines are for qualification of the original data based on the confirmation data obtained by LC/MS/MS and apply to HE by SW-846 Method 8330B and perchlorate by EPA Method 314. It should be noted that no confirmation LC/MS/MS results are qualified. If the original sample result is a detect, and the corresponding LC/MS/MS result is a detect, the original result is considered to be confirmed, although confirmation qualifiers may be applied. As with all validation guidelines, professional judgment is the final criteria.

4.5.1 Required LC/MS/MS Data

The laboratory is expected to include all calibration and QC data normally supplied in a level 4 data package; however, only the following information is necessary to evaluate the confirmation.

1) Form I Sample Results
2) CRI Summary
3) CCV %D Summary
4) IS Recovery
5) MB results

4.5.2 LC/MS/MS QC are Acceptable

The laboratory is expected to meet all QC acceptance criteria when performing confirmation by LC/MS/MS. **Note:** Multiple QC failures should result in “NJ” or no qualification to original results.

If all LC/MS/MS QC acceptance criteria are met:

Evaluation	**Action**
If the original result is ≥ the PQL and the LC/MS/MS result is non-detect,	qualify the original result as "R."
If the original result is < the PQL and the LC/MS/MS result is non-detect,	qualify the original result as "R."
If the original result and the LC/MS/MS result are > the PQL, and… the %D is >40%,	 qualify the original result as "J."
If the original result is ≥ the PQL and the LC/MS/MS result is < the PQL,	qualify the original result as "NJ+."
If the original result is < the PQL and the LC/MS/MS result is ≥ the PQL,	qualify the original result as "J-."

4.5.3 Method Blank

The MB performed on LC/MS/MS should be < the MDL.

Evaluation	**Action**
If the LC/MS/MS MB concentration is ≥ the MDL and…	
the original result is > the PQL, but <5X the MB concentration,	qualify the original result as "R."
the original result is < the PQL and <5X the MB concentration,	qualify the original result as "NJ+."

4.5.4 Continuing Calibration

If the original result is a detect and the LC/MS/MS result is a non-detect and the LC/MS/MS CCV is outside acceptance criteria:

Evaluation	**Action**
If the CCV %D is positive (high bias), >20% and…	

Evaluation (concluded)	Action (concluded)
the original result is ≥ the PQL,	qualify the original result as "R."
the original result is < the PQL,	qualify the original result as "R."
If the CCV %D is negative (low bias), >20% and…	
the original result is ≥ the PQL,	qualify the original result as "R."
the original result is < the PQL,	qualify the original result as "NJ."

4.5.5 PS/CRI

If the original result is a detect, the LC/MS/MS result is a non-detect, and the LC/MS/MS PS/CRI are outside acceptance criteria:

Evaluation	Action
If the spike %R is > the upper limit and…	
the original result is ≥ PQL,	qualify the original result as "R."
the original result is < PQL,	qualify the original result as "R."
If the spike %R is < the lower limit and…	
the original result is ≥ the PQL,	qualify the original result as "R."
the original result is < the PQL,	qualify the original result as "NJ."

4.5.6 IS Performance

In the case of confirmation analysis, IS performance is assessed only as it reflects instrument sensitivity, not calculated bias; and only applies if the IS is used for quantification.

If the original result is a detect; the LC/MS/MS result is a non-detect; and the IS is outside acceptance criteria:

Evaluation	Action
If the IS %R is > the upper limit and…	
the original result is ≥ the PQL,	qualify the original result as "R."

Evaluation (concluded)	Action (concluded)
the original result is < the PQL,	qualify the original result as "R."
If the IS %R is < the lower limit and…	
the original result is ≥ the PQL,	qualify the original result as "R."
the original result is < the PQL,	qualify the original result as "NJ."

4.6 Procedure for Inorganic Data Validation

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for target analytes. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run, and CCV documents that the ICAL is still valid.

4.6.1 Initial Calibration

Criteria: Instruments used for all analyses except ion chromatography (IC) must be calibrated daily and each time the instrument is set up as noted below.

Inductively coupled plasma-atomic emission spectroscopy (ICP)-AES) radial-viewing analysis: A blank and at least one standard must be used in establishing the analytical curve. **ICP-AES axial-viewing analysis:** A blank and at least three standards must be used in establishing the analytical curve.

This requirement specifically addresses the trace analysis of arsenic, cadmium, lead, antimony, selenium, and thallium using axial-viewing ICP-AES instead of graphite furnace atomic absorption for samples with analyte concentrations below 500 parts per billion (ppb).

Inductively coupled plasma-mass spectrometry (ICP-MS) analysis: A blank and at least one standard must be used in establishing the analytical curve.

Mercury analysis by cold vapor atomic absorption: A blank and at least four standards must be used in establishing the analytical curve.

Cyanide analysis: A blank and at least three standards, one of which must be at the PQL, must be used in establishing the analytical curve.

IC analysis: A blank and at least three standards, one of which must be at the PQL, must be used in establishing the analytical curve. Daily calibration is not required if acceptable calibration verification is performed prior to the analytical run.

Flow Injection and Colorimetric analysis: A blank and at least three standards, one of which must be at the PQL, must be used in establishing the analytical curve.

Evaluation	Action
If the minimum number of standards was not used for ICAL,	qualify all detects as "J" and all non-detects as "UJ."
If the instrument was not calibrated at the proper frequency,	qualify all sample results as "R."
If only one standard was used for trace axial view ICP-AES,	notify the laboratory and the program that the laboratory was not compliant with the contract SOW.

Criteria: The correlation coefficient (r) of the ICAL curve shall be ≥0.995, and the absolute value of the intercept shall be ≤3X the MDL.

Note: The sample results may be reported with non-detects at the MDL or at the PQL value. See below for appropriate evaluation.

The r assessment need only be performed on those curves established using at least three standards and a blank (four-point curve).

The intercept shall be assessed for all inorganic calibration curves, with the following exception: The laboratory may report two calibration lines for some inorganic analytes. In this case, qualifiers should be applied if the r criteria are not met for either reported calibration. The intercepts for these curves should not be evaluated.

Evaluation	Action
If any compound has a r:	
<0.995 but ≥0.90,	qualify all associated detects as "J" and all associated non-detects may be qualified as "UJ" if any other calibration criteria have been exceeded for that analyte.
<0.90 but ≥0.80,	qualify all associated detects as "J" and all associated non-detects as "UJ."
<0.80,	qualify all associated detects as "J" and all associated non-detects as "R."

Evaluation (concluded)	**Action (concluded)**
When results are reported at the MDL: If the intercept for any target compound is negative with an absolute value... > the MDL but ≤3X the MDL, >3X the MDL,	 qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ." qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "R."
If any compound has an intercept that is positive and > the MDL,	qualify all associated detects <3X the intercept as "J+."
When results are reported at the PQL: If the intercept for any target compound is negative with an absolute value... > the MDL but ≤2X the PQL, >2X the PQL,	 qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ." qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "R."

4.6.2 CCV

Criteria: ICV and CCV: An ICV standard must be analyzed after instrument calibration and prior to sample analysis. A CCV standard must be analyzed once every 10 injections or every two hours, whichever is more frequent. The evaluation of CCV data applies to all CCVs that bracket samples of interest.

ICV and CCV analysis results must be within the recovery acceptance criteria of 90% to 110% of the true value for all analytes except mercury and cyanide.

ICV and CCV analysis results for mercury must fall within the recovery acceptance criteria of 80% to 120% of the true value.

ICV and CCV analysis results for cyanide must fall within the recovery acceptance criteria 85% to 115% of the true value.

Evaluation	Action
If the ICV and CCV standards were not analyzed at the proper frequency, or if either a required ICV or CCV was not analyzed, or if all target compounds were not present in any ICV or CCV standard,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If all required ICVs and CCVs were not analyzed,	qualify all associated detects as "J" and all associated non-detects as "R."
If the ICV or CCV %R is…	
<90% but ≥75% (mercury: <80% but ≥65% and cyanide: <85% but ≥70%),	qualify all associated detects as "J-" and all associated non-detects as "UJ."
>110% but ≤125% (mercury: >120% but ≤135%, cyanide: >115% but ≤130%),	qualify all associated detects as "J+."
<75% (mercury: <65%, cyanide: <70%),	qualify all associated detects "J-" and all associated non-detects as "R."
>125% (mercury: >135%, cyanide: >130%),	qualify all associated detects as "R."

4.6.3 Blanks

Blank analysis results are assessed to determine the existence of contamination problems. The criteria for evaluation of blanks apply to any laboratory blank associated with the samples. See Section 4.1.2 for general chemistry QC exemptions.

Criteria: An ICB must be analyzed to verify the baseline immediately following calibration and prior to analytical sample analysis. A CCB must be analyzed after each CCV and at the end of every analytical sequence in order to bracket all sample runs. All CCBs that bracket samples of interest shall be reported and assessed.

A minimum of one MB (or preparation blank) should be analyzed for every 20 samples. The same reagents used for the sample digestion must be used to prepare the MB. In those cases for which reagents are automatically added to all samples by an autoanalyzer, the ICB is equivalent to a preparation blank. FBs and EBs are treated as preparation blanks.

If any QC problems exist with any blank, all data associated with the batch must be evaluated to determine whether there is an inherent variability in the data for the batch, or if the problem is an anomaly not affecting other data.

If the absolute value of the ICB or CCB result is > the PQL, the analysis should have been terminated and the problem corrected by the laboratory. If any analyte concentration in the blank is > the PQL, the lowest reported concentration in the associated samples must be ≥10X the concentration in the blank. Samples having analyte concentrations <10X that of the blank but > the PQL shall be re-digested and/or reanalyzed.

No contaminants ≥ MDL should be present in the blanks.

When there is blank contamination and reanalysis is not possible, the data may need to be qualified. Use the blank (ICB, CCB, MB, FB, or EB) with the highest concentration associated with the samples of interest to qualify data. If a CCB is used to qualify data, it must bracket the sample of interest.

The effect of MB values versus ICB/CCB values on sample results is not straightforward and will vary depending on analytical method. Professional judgment is required to properly assess the effect of blank data on sample results. As a general guideline, in the case of conflicting positive and negative MB and ICB/CCB values, the MB values will take precedence over ICB/CCB values when applying qualifications to associated sample results.

Evaluation	**Action**
If blank frequency criteria were not met,	note the deficiency in the data validation report.
If any associated blank value is positive and is ≥ the MDL but ≤ the PQL,	qualify all associated detects ≥ the MDL but <5X blank value as "U" at 5X the blank value. All associated detects >5X blank may be qualified "J+" based on professional judgment.
If any ICB/CCB value is positive and > the PQL,	qualify all associated sample results < the PQL as "U" at the PQL and all associated detects > the PQL but <5X the blank value as "UJ" at 5X the blank value or as "R" based on professional judgment. All associated detects >5X blank value may be qualified "J+" or "R" based on professional judgment.
If any MB, FB, or EB value is positive and > the PQL,	qualify all associated detects <5X the blank value and all associated non-detects as "UJ" at 5X the blank value. All associated detects >5X the blank value may be qualified "J+" or "R" based on professional judgment.

Evaluation (concluded)	Action (concluded)
If the absolute value of the negative blank is > the MDL but ≤ the PQL,	qualify all associated detects <5X the MDL as "NJ-" and all associated non-detects as "UJ."
If the absolute value of a negative ICB/CCB value is > the PQL, and the analysis was not terminated by the laboratory,	notify the laboratory and qualify all associated detects <10X the PQL as "NJ-" or "R" and all associated non-detects as "R."
If the absolute value of the negative MB, FB, or EB value is > the PQL, and the analysis was not terminated by the laboratory,	notify the laboratory and qualify all associated detects <5X the blank value as "UJ" or "R" based on professional judgment and all associated non-detects as "R." Sample results >5X the blank value may be qualified as "J-" or "R" based on professional judgment.

4.6.4 MS

The MS sample analysis is performed as a measure of the ability to recover analytes in a particular matrix.

Criteria: The MS data shall not be used to evaluate data unless the MS sample was from the same client and of similar matrix.

An MS sample shall be analyzed at a frequency of once per data package, once per 20 samples of similar matrix, or once per sample matrix, whichever is more frequent.

Samples identified as FBs and EBs cannot be used for MS analysis.

Spiking levels shall be approximately at the mid-point of the calibration range.

The MS recovery acceptance criteria are 75% to 125%, unless the sample concentration is >4X the spike concentration (see Section 4.1.20).

MS analysis shall be performed for all analytes other than sodium, potassium, magnesium, and calcium.

For methods, which require a digestion, PSs are occasionally performed. The recovery acceptance criteria on a PS are 85% to 115%. For methods which do not require digestion (i.e. IC, ion-specific electrode, and colorimetric techniques), MSs shall be analyzed. These spikes may be referred to as a post spikes or analytical spikes. These should be evaluated using the recovery acceptance criteria of a MS, 75% to 125%.

Evaluation	Action
If the MS sample was from another client or of a dissimilar matrix, the frequency of the MS did not meet specified criteria, an MS was not analyzed, or an FB or EB was used for MS analysis,	qualify all detects as "J" and all non-detects as "UJ."
If an MS %R is…	
>125%,	qualify all associated detects as "J+."
<75% but ≥30%,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
<30%,	qualify all associated detects as "J-" and all associated non-detects as "R."
If an MS/MSD pair was analyzed and recoveries of any target analyte are both above and below acceptance criteria,	qualify all detects as "J" and all non-detects as "UJ."
If the PS %R is…	
>115%,	qualify all associated detects as "J+."
If the PS %R is < the acceptance criteria,	qualify all associated detects as "J-" and all associated non-detects as UJ" if the recovery is ≥10% and as "R" if the recovery is <10%.

4.6.5 Replicate

Replicate analyses are indicators of laboratory precision based on each sample matrix.

Criteria: One replicate must be analyzed for each matrix or each batch, with a minimum frequency of one per 20 samples.

Samples identified as FBs or EBs should not be used for replicate or MSD analysis.

An acceptance limit of 20% for the RPD shall be used for sample values ≥5X the PQL. For solid and waste samples, it may be appropriate to accept an RPD of up to 35% based on professional judgment.

A control limit of ± the PQL shall be used for sample values > the PQL but <5X

the PQL, including the case when only one of the replicate sample values is > the PQL but <5X the PQL.

No precision criteria apply when both replicate sample values are < the PQL.

When a replicate was not performed but an MSD was analyzed, the MS/MSD RPDs are evaluated as specified in Section 4.3.5. If neither a replicate nor an MS/MSD were analyzed, the laboratory may run an LCSD to measure precision. LCS/LCSD RPDs are evaluated as specified in Section 4.3.5.

Evaluation	**Action**
If no replicate sample, MSD, or LCSD were analyzed for each matrix or for each data package, or if an FB or EB was used for the replicate analysis,	qualify all detects of the same matrix as "J" and all non-detects of the same matrix as "UJ."
If the original result and replicate result are both ≥5X the PQL and the RPD exceeds the appropriate control limit,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."
If the original and/or replicate result is > the PQL but <5X the PQL (including non-detects) and the difference between the original result and replicate result is > the PQL,	qualify all associated detects of the same matrix as "J" and all associated non-detects of the same matrix as "UJ."

4.6.6 LCS

Data for LCSs are generated to provide information on the accuracy of the analytical method and the overall laboratory performance, including sample preparation.

Criteria: LCSs shall be analyzed using the same sample preparation and analysis methods used for samples, with one LCS analyzed with each batch of up to 20 samples.

Multiple LCS analyses may not be used to meet acceptance criteria; that is, if multiple LCSs are analyzed for a batch and any failures occur, the failed LCS will be used to qualify the data. For all aqueous LCS results, the recovery acceptance criteria are 80% to 120%, except antimony and silver. The recovery acceptance criteria for silver and antimony are laboratory-specified. LCS failures for silver and antimony shall be discussed in the data validation report but shall not be subject to the reanalysis requirement.

For all solid LCS results, the recovery acceptance criteria are established by the agency that prepared the reference material or statistically-derived criteria developed by the laboratory. The laboratory should report these acceptance

criteria on the LCS reporting form. If solid LCS acceptance criteria are not provided, then 30% to 150% should be used to assess soil results. If this situation occurs, it should be noted in the data validation report. A solid LCS should be analyzed for mercury in solid analyses.

An aqueous LCS is not required for mercury or cyanide analyses. Since the ICV is always digested/distilled for these analyses, it is equivalent to an LCS.

Evaluation	**Action**
If an LCS was not analyzed,	qualify all detects as "J" and all non-detects as "UJ."
<u>Aqueous LCS</u> If the LCS %R is…	
>120%,	qualify all associated detects as "J+."
<80% but ≥50%,	qualify all associated detects as "J-" and all associated non-detects "UJ."
<50%,	qualify all associated detects as "J-" and all associated non-detects as "R."
<u>Solid LCS</u> If the LCS %R is…	
> the upper control limit,	qualify all associated detects as "J+."
≥30% but < the lower control limit,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
<30%,	qualify all associated detects as "J-" and all associated non-detects as "R."
If an LCS/LCSD pair was analyzed and recoveries for any target analyte are both above and below acceptance criteria,	qualify all detects as "J" and all non-detects as "UJ."
If an aqueous LCS was analyzed for soil matrices,	qualify all detects as "J" and all non-detects as "UJ."

4.6.7 RLV

Criteria: RLV standards (i.e., RLV for atomic absorption [AA] methods [CRA], RLV for cyanide methods [CRDL], and CRI) are analyzed at the beginning of each analytical run as a measure of accuracy near the RL. CRA and CRDL standards

are prepared with concentrations at the PQLs, and CRI standards are prepared with concentrations at twice the PQLs.

The advisory recovery acceptance criterion for these analyses is 70% - 130%.

Evaluation	Action
If the RLV recovery is…	
>130%,	qualify all associated detects <5X the PQL as "J+."
<70% but ≥30%,	qualify all associated detects <5X the PQL as "J-" and all associated non-detects as "UJ."
<30%,	qualify all associated detects <5X the PQL as "J-" and all associated non-detects as "R."

4.6.8 Method-specific analytical requirements (inorganic)

4.6.8.1 ICP-AES and ICP-MS Methods

ICS

The ICP-AES and ICP-MS ICSs (interference check sample solution A (ICS A) and interference check sample solution AB (ICS AB)) verify the instrument's interelement and background correction factors.

Criteria: An ICS A must be analyzed at the beginning of each sample analysis run.

Absolute values for all ICS A target analytes, except those in the ICS A solution, must be ≤ the MDL.

Evaluation	Action
If the ICS A sample was not analyzed at the required frequency,	note the deficiency in the data validation report.
If the sample concentrations of aluminum, calcium, iron, and/or magnesium are < their respective concentrations in the ICS A solution,	accept the sample results without qualification.

Evaluation (concluded)	**Action (concluded)**
If the sample concentrations of aluminum, calcium, iron, and/or magnesium are comparable to or > their respective concentrations in the ICS A solution, and the ICS A result for a non-spiked analyte is...	
positive and ≥ the MDL,	qualify all associated sample detects < 50X the ICS A result as "J+."
negative and the absolute value of the result is > the MDL but ≤2X the MDL,	qualify all associated detects <50X the absolute value of the ICS A result as "J-" and all associated non-detects as "UJ."
negative and the absolute value of the result is >2X the MDL,	qualify all associated detects <50X the absolute value of the ICS A result as "J-" and all associated non-detects as "R."

Criteria: An ICS AB must be analyzed at the beginning of each sample analytical run.

ICS AB results for the target analytes in the ICS AB solution must be within 80% to 120% of the true value.

If the recovery criteria are not met, the analyst may either terminate the analysis or continue and re-analyze the failed constituents at a later time.

Evaluation	**Action**
If the ICS AB was not analyzed at the required frequency,	note the deficiency in the data validation report.
If the concentrations of aluminum, calcium, iron, and magnesium in the sample are < their respective concentrations in the ICS AB solution,	accept the sample results without qualification.
If the sample concentrations of aluminum, calcium, iron, and magnesium are comparable to or > their respective concentrations in the ICS AB solution and the ICS AB recovery for an analyte is...	
>120%,	qualify all associated detects as "J+."

Evaluation (concluded)	Action (concluded)
<80% but ≥50%,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
<50%,	qualify all associated detects as "J-" and all associated non-detects as "R."

ICP Serial Dilution (SD)

The ICP SD monitors physical or chemical interferences that may exist in each sample matrix.

Criteria: A SD must be analyzed for each matrix in an analytical run. If the undiluted results for the sample used for SD are ≥50X the MDL, then the %D between a 5X dilution result and the original result must agree within 10%.

Samples with elevated concentrations that require dilutions >50X or that require multiple dilutions must also meet these requirements. However, care should be used in evaluating the result from the undiluted sample since it may be above the linear range of the instrument and would not apply.

No acceptance criterion applies when the undiluted sample result is <50X the MDL.

Evaluation	Action
If frequency requirements are not met,	qualify all detects ≥50X the MDL as "J."
If the result for any analyte in the sample used for SD analysis is ≥50X the MDL and the %D is >10%,	qualify detects for all samples of the same matrix in the batch as "J" and non-detects all samples of the same matrix in the batch as "UJ."

Instrument Tuning for ICP-MS

Tuning and performance criteria are established to ensure mass resolution and identification. These criteria are not sample specific. Conformance is determined using standards. Therefore, these criteria should be met in all circumstances.

Criteria: The ICP-MS tune shall be evaluated daily. The tuning solution must contain elements representing all of the regions of interest. The mass calibration must be within 0.1 atomic mass units (amu) of the true value. The resolution must be verified to be <0.9 amu full width at 10% peak height.

Evaluation	Action
If tunes were not run daily or if all mass calibration and resolution criteria were not met,	use professional judgment to determine which data should be used. It is suggested that all associated detects should be qualified as "J" and all associated non-detects should be qualified as "UJ."
If multiple QC failures also occurred,	qualify all results as "R."

IS Performance for ICP-MS

IS criteria ensure that ICP-MS sensitivity and response are stable and acceptable during each analysis. They also allow for monitoring of indigenous quantities of the ISs.

Criteria: The intensity of the IS in the samples must fall within 60% to 125% of the intensity of the IS in the ICAL standard. The intensity of the IS in the bracketing CCVs and CCBs must fall within 80% to 120% of the intensity of the IS in the ICAL standard.

Evaluation	Action
If no IS was used,	qualify all results as "R."
If the IS intensity for a sample is…	
≥30% but <60% of the intensity of the IS in the calibration standard,	qualify all associated detects as "J+" and all associated non-detects as "UJ."
>125% but ≤160% of the intensity of the IS in the calibration standard,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
<30% or >160 % of the intensity of the IS in the calibration standard,	qualify all associated results as "R."
If both the CCV and CCB have IS intensities outside of the recovery limits,	all associated sample results may be qualified as "J/UJ" due to instrument drift based on professional judgment.

4.6.8.2 Total Organic Carbon (TOC) by SW-846 Method 9060

Criteria: Quadruplicate analyses are required. The average is to be reported.

Evaluation	Action
If quadruplicate analyses were not run,	qualify all detects as "J" and all non-detects as "R."

4.6.8.3 Total Cyanide and Cyanide Amenable to Chlorination

After evaluation of total cyanide data (and application of appropriate qualifiers) using Sections 4.6.1 through 4.6.7, proceed to the following guidance for further evaluation of analytical data for total and amenable cyanide,

Criteria: Sample preparation includes distillation of the samples. In addition to the field samples, the QC samples and one standard and the ICV must be distilled. The LCS meets the requirement for distillation of one standard if the concentrations of the LCS and ICV are different.

Evaluation	Action
If the samples, appropriate QC samples, and appropriate standards were not distilled,	qualify all results as "R."
If the field samples were distilled but the QC samples and/or standards were not distilled,	qualify all associated detects as "J" and all associated non-detects as "UJ."

The remainder of this section is provided as guidance for the assessment of data for cyanide amenable to chlorination. Total cyanide data are to be reviewed according to the guidance in Sections 4.6.1 through 4.6.6.

Cyanide amenable to chlorination (decomposed by chlorination) is derived by measuring the total cyanide and the cyanide remaining after chlorination. The amenable cyanide is calculated as the difference between the total and chlorinated. Biases in the cyanide after chlorination will result in a bias in the opposite direction for the calculated amenable result.

The actual analysis of the sample is the same for the total and the chlorinated analysis; only the sample preparation is different. The laboratory will generally run the total and the chlorinated samples together in the same batch and will use the same calibration and calibration checks for both the total and chlorinated cyanide samples. The laboratory should identify which sample is the total sample and which sample is the chlorinated sample.

LCS and MS solutions are generally of the form that will not decompose with chlorination giving a %R of zero for chlorinated cyanide. When the chlorinated cyanide recovery is 0%, the reported amenable cyanide recovery is equal to the total cyanide recovery. Alternatively, the LCS and MS

solutions may not decompose with chlorination and will give a recovery of 0% for amenable cyanide. The laboratory should discuss in the case narrative the form of solution that was used for LCS and MS analyses. The chlorinated cyanide LCS and MS recoveries may not be reported. In this case, the chlorinated cyanide LCS and MS recoveries can be determined from the raw data. The amenable cyanide LCS and MS data should be evaluated using the criteria given for total cyanide.

When total and amenable cyanide are analyzed together in the same run, all initial and continuing calibration qualification applied to the total cyanide results should also be applied to the amenable cyanide results. When total and chlorinated cyanide are not analyzed in the same run, qualifications applied to the total and chlorinated cyanide results should also be applied to the amenable cyanide results. Signed qualification for chlorinated cyanide results should be reversed for amenable cyanide results; that is, a "J+" for chlorinated cyanide results would be a "J-" for the associated amenable cyanide.

Major differences in total and chlorinated results are generally attributed to incomplete destruction of cyanide complexes such as thiocyanide. Non-detects for total cyanide with significant cyanide results for chlorinated cyanide (negative amenable cyanide) may indicate a significant presence of thiocyanide or other cyanide complexes in the sample.

Criteria: A CCV standard must be analyzed once every 10 injections or every two hours, whichever is more frequent. The evaluation of CCV data applies to all CCVs that bracket samples of interest.

The recovery acceptance criteria for CCV analysis results must be within 85% to 115%.

Evaluation	**Action**
If a chlorinated cyanide CCV %R is…	
<85% but ≥70%,	qualify all associated detects for amenable cyanide as "J+."
>115% but ≤130% and the amenable cyanide result is < the total cyanide result,	qualify all associated detects for amenable cyanide as "J-" and all associated non-detects for amenable cyanide as "UJ."
<70%,	qualify all associated detects for amenable cyanide as "R."
>130% and the amenable cyanide result is < the total cyanide result,	qualify both detects and non-detects for amenable cyanide as "R."

Criteria: A minimum of one MB should be analyzed for every 20 samples. The same reagents used for the sample must be used to prepare the MB. A CCB must be analyzed after each CCV and at the end of every analytical sequence. All CCBs that bracket samples of interest shall be reported and assessed.

If cyanide is detected in an MB or CCB, the chlorinated sample results must be assessed to determine the impact on amenable cyanide results.

No contaminants ≥ the MDL should be present in the blanks.

Evaluation	**Action**
If a chlorinated cyanide MB or CCB value is positive and ≥ the MDL and the chlorinated sample result is a detect <5X the MB/CCB value,	qualify all associated detects for amenable cyanide as "J-"and, if the total cyanide result is > the MDL, qualify all associated non-detects for amenable cyanide as "UJ."
If the absolute value of the negative chlorinated cyanide MB or CCB value is > the MDL and the chlorinated sample result is < 5X the MDL or non-detect,	qualify all associated detects for amenable cyanide <5X the MDL as "J+."

Criteria: The absolute value of a negative amenable cyanide result must be <3X the MDL.

Note: Laboratories will generally report the amenable cyanide as non-detect when the chlorinated result is > the total cyanide result. The raw data may need to be reviewed to determine the actual negative amenable cyanide result.

Evaluation	**Action**
If the absolute value of a negative amenable cyanide result is >3X the MDL and the total cyanide is a non-detect,	note in the data validation report but do not qualify any results.
If the absolute value of a negative amenable cyanide result is >3X the MDL and the total cyanide is a detect,	qualify the amenable cyanide result as "UJ" if the absolute value of the amenable cyanide result is >3X but ≤10X the MDL and as "R" if the absolute value of the amenable cyanide result is >10X the MDL.

4.6.8.4 Total/Partial Inorganic Analyte Results

Several inorganic analytes are analyzed and reported as total and partial results, (i.e. total chromium/hexavalent chromium, total kjeldahl nitrogen (TKN)/ ammonia, hardness/calcium and magnesium, total alkalinity/carbonate and bicarbonate, total cyanide/amenable cyanide, and total

phosphorus/phosphate. In these cases, it is expected that the partial value will be ≤ the total value. These reported values may or may not be obtained from the same analytical method. When the reported result for the partial analyte is > the result for the total analyte, one or both results are suspect. The extent the quality of the data is affected must be determined. The following criteria should be used for guidance.

Note: Comparisons are made at the elemental level, that is, total nitrogen should be > the nitrogen in ammonia not > the ammonia.

Criteria: When both a partial and a total result are reported, the result for the partial analyte must be ≤ the result for the total analyte.

If the partial result is > the total result, the laboratory should be contacted for further information. If the laboratory cannot be contacted or cannot provide sufficient explanation, the following criteria apply.

If the partial result is > the total result and both results are ≥5X the PQL, then the RPD between the two values should be ≤20%.

If the partial result is > the total result and one or both results are <5X the PQL, then the difference between the two values should be ≤ the partial analyte's PQL.

Evaluation	**Action**
If the partial result is > the total result, and…	
both the total and partial results are ≥5X the PQL and the RPD is >20%,	may qualify one or both results as "R" or "J" based on professional judgment.
one or both results are <5X the PQL and the difference between the two results is > the partial analyte's PQL,	may qualify one or both results as "R" or may qualify all associated detects as "J" (with or without bias) or may qualify all associated non-detects as "UJ" based on professional judgment.

Partial Analyte Conversions

To Convert	To	Multiply By
O-phosphate	Phosphorus	0.326
Ammonia	Nitrogen	0.824
Ca Mg	Hardness Hardness (total hardness is the sum of the calculated Ca and Mg hardness results)	2.497 (2.5, if titrated) 4.118 (4.12, if titrated)

Alkalinity Relationships

The results obtained from the phenolphthalein and total alkalinity determinations offer a mean for stoichiometric classification of the three principal forms of alkalinity present in many waters.

- Carbonate alkalinity is present when the phenolphthalein alkalinity is not zero and is not < the total alkalinity.
- Hydroxide alkalinity is present if the phenolphthalein alkalinity is > half the total alkalinity.
- Bicarbonate alkalinity is present if the phenolphthalein alkalinity is < half the total alkalinity.

Result of Titration	Hydroxide Alkalinity as $CaCO_3$	Carbonate Alkalinity as $CaCO_3$	Bicarbonate Alkalinity as $CaCO_3$
P=0	0	0	T
P< ½ T	0	2P	T-2P
P= ½ T	0	2P	0
P> ½ T	2P-T	2(T-P)	0
P=T	T	0	0

P- phenolphthalein alkalinity; T- total alkalinity

Phenolphthalein alkalinity is the term traditionally used for the quantity measured by titration to pH 8.3. It is not routinely reported by the laboratories but could be calculated using the amount of titrant used to reach pH 8.3. There is usually a column on the alkalinity worksheet that contains this information. Since total alkalinity is reported, it should be verified that the carbonate, bicarbonate, and hydroxide values do not exceed the total.

No conversion is required to compare hexavalent chromium to total chromium.

The calculation for amenable cyanide is detailed in Section 4.6.8.3.

4.6.8.5 Data Validation for Analyses by NIOSH Method 7300

This procedure is for the analysis of elements capable of detection by ICP-AES analysis, including Be, in air samples. The samples are collected onto filters at a flow rate of 1 to 4 L/min. The working range of this method is 0.005 to 2.0 milligrams (mg) per cubic meter (2.5-1000 ug/sample) for each element in a 500-L air sample.

The laboratory must follow the requirements specified in NIOSH 7300 as well as any requirements specified in the AHIA Accreditation Requirements. Compliance requirements for satisfactory data reporting include:

- Case narrative
- Initial calibration data
- Continuing calibration data

- Media blanks, FBs, and preparation blank data
- LCS/LCSDs
- Sample results
- Instrument run logs

Initial Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and CCV documents that the ICAL is still valid.

Criteria: Calibrate the spectrometer according to the manufacturer's recommendations. Typically, an acid blank and a 10 ug/mL multi-element***** working standard is used.

*****refer to method for chemically compatible combinations of elements

Evaluation	**Action**
If the minimum number of standards as defined in the criteria section was not used for ICAL,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If the instrument was not calibrated as required,	qualify all associated detects and all associated non-detects as "R."

Calibration Verification

Criteria: A CCV standard must be analyzed for every 10 samples. The recovery acceptance criteria for analysis results must be within the 90% to 110% of the true value for all analytes.

Evaluation	**Action**
If the CCV frequency criteria were not met	qualify all associated detects as "J" and all associated non-detects as "R."
If the ICV or CCV %R is…	
≥75% but <90%,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
≥110% but <125%,	qualify all associated detects as "J+."

Evaluation (concluded)	Action (concluded)
If the ICV or CCV %R is…	
<75%,	qualify all associated detects as "J-" and all associated non-detects as "R."
>125%,	qualify all associated detects as "R."

Blanks

Blank analysis results are assessed to determine the existence of contamination. During sampling, two to ten FBs are collected per sample set. During sample preparation a reagent blank (MB) and media blanks are included with the samples during the digestion process. The average media blank result (ug/mL) is subtracted from the sample result (ug/mL) in the final calculation. In instances where more than one blank (FB or MB) associated with a given sample is ≥ the MDL, qualification is to be performed using the associated blank with the highest concentration of contaminant.

Criteria: The same reagents used for the sample digestion must be used to prepare the MB. No contaminants > the MDL may be present in the blanks.

Evaluation	Action
If the frequency criteria were not met,	note the deficiency in the data validation report.
If problems with any blank exist,	all data associated with the batch must be evaluated to determine whether there is an inherent variability in the data for the batch or if the problem is an anomaly not affecting other data.
If any analyte concentration in the blank is in excess of the PQL,	the lowest reported concentration in the associated samples must be > 10X the concentration in the blank or results must be qualified or rejected.
If a blank value is ≥ the MDL,	qualify all associated detects <5X the blank concentration as "J."

LCS/LCSD

The LCS/LCSD serves as a measure of the overall performance of all steps in the analysis, including sample preparation.

Criteria: The LCS/LCSD must be analyzed using the same preparation and analysis methods used for samples, with one LCS/LCSD analyzed for each batch of up to 10 samples.

All LCS/LCSD results for air filters must fall within the recovery and RPD control limits established by the agency that prepared the reference material or by statistically-derived limits developed by the laboratory. The laboratory is to include these limits on the LCS/LCSD reporting form.

Professional judgment may be used to determine the need for qualification of sample results based on whether or not the LCS, LCSD, or both meet QC acceptance criteria.

Evaluation	**Action**
If the LCS/LCSD was not analyzed,	qualify all associated detects as "J" and all associated non-detects as "UJ."
If the %R is…	
> upper control limit,	qualify all associated detects as "J+."
>30% and < the lower control limit,	qualify all associated detects as "J-" and all associated non-detects as "UJ."
<30%,	qualify all associated detects as "J-" and all associated non-detects as "R."
If RPD criteria were not met,	qualify detects for associated compounds as "J" and non-detects as "UJ.

Dilutions

The PQLs must be adjusted to reflect all sample dilutions. Original undiluted results document the actual MDLs for non-detected compounds.

Criteria: It must be determined that the laboratory strove to make dilutions in such a way that the final concentration was measured in the mid-range of the calibration curve, and that the laboratory did not report results from measurements above the highest concentration standard.

Evaluation	**Action**
If any samples required dilution because one or more analytes exceeded the calibration range, and…	

Evaluation (concluded)	Action (concluded)
the original undiluted results were reported,	qualify all associated detects > the high standard as "J."
only the diluted results were reported,	qualify all associated non-detects as "UJ."

4.6.8.6 Method 300.0/9056A, Determination of Inorganic Anions by Ion Chromatography

Criteria: RTs for ICV and CCV analyses must be within 30 seconds (0.5 minutes) of those in the ICAL midpoint.

Evaluation	Action
If any target analyte RT in the CCV varies by more than ±30 seconds from that of the associated ICAL standards,	qualify all associated detects as "J" and all associated non-detects as "UJ."

4.7 Procedure for Radiochemical Analyses Validation

4.7.1 Quantification

Criteria: Radiochemical analytical results shall be reported as measured and shall include the TPU at the 95% confidence level (2-σ).

Note: Some programs may request the result be reported with the 1-σ uncertainty. When this is the case the reported uncertainty must be multiplied by two for evaluation of quantitation and replicate error ratio (RER).

The laboratory shall report all results regardless of concentration or sign and shall not report any result as "less than."

The laboratory shall include a sample minimum detectable activity (MDA) calculated using sample-specific parameters.

For programs that require application of one final qualifier to sample results, if the "BD" qualifier is applied to a sample result, the result shall not be further qualified as "J" due to other QC failures.

Note: Some programs may request the "U" qualifier instead of the "BD" qualifier.

Extremely large errors/uncertainties may indicate inappropriate error calculation. If large errors/uncertainties are reported with the results, the laboratory should verify the calculations.

Evaluation	Action
If the sample result is < the 2-σ TPU,	qualify the result as "BD."
If the sample result is < the MDA,	qualify the result as "BD."
If the sample result is ≥ the MDA but <3X the MDA,	qualify the result as "J."
If the absolute value of a negative result (excluding gamma spectroscopy, addressed below) is > the MDA,	qualify the result as "R."
Gamma Spectroscopy: If the absolute value of a negative sample result is >2X the MDA,	qualify the result as "R."

If the "BD" qualifier is applied to a sample result, the result shall not be further qualified as "J" due to other QC failures.

4.7.2 Blanks

Blank analysis results are assessed to identify contamination. The criteria for evaluation of blanks apply to all blanks associated with the samples.

Criteria: One MB (or preparation blank) must be analyzed for each matrix and each batch, or for every 20 samples, whichever is most frequent.

MB analysis is required for all analyses requiring sample preparation.

Samples associated with any preparation blank result that is ≥ the MDA shall be redigested and reanalyzed. Exceptions to this requirement are samples for which the measured sample concentration is ≥10X the preparation blank value.

Evaluation	Action
If the prep blank was not analyzed at the proper frequency and there are sample concentrations ≥ the MDA but <5X the MDA,	qualify those results as "J."
If the blank result is positive and is statistically >0.0 (i.e., > the 2-σ TPU and ≥ the MDA),	qualify all associated sample results ≥ the MDA but <5X the blank value as "NJ+."

Evaluation (concluded)	Action (concluded)
If the absolute value of a negative blank result is > the MDA,	qualify all associated detects ≥ the MDA but <5X the MDA as "NJ-." The program may require results < the MDA to be qualified "UJ."
If the absolute value of a negative blank result is >5X the MDA,	notify the laboratory and qualify all associated sample results as "R."
If the absolute value of the negative blank result is > 5X the MDA for liquid scintillation analyses such as tritium, where the calibration blank is subtracted from the result,	notify the laboratory and qualify all sample results <5X the MDA as "R."

4.7.3 Sample-Specific Chemical/Tracer Recovery

An addition of a known quantity of radioactive or chemically similar material to a sample prior to chemical separation is used to determine the amount of the analyte recovered.

Criteria: Recovery guidelines for tracer and carrier results shall be 50% to 105%. Optionally, low tracer recoveries may be evaluated from the total area counts. Samples with low recoveries but with tracer area counts >400 counts may or may not be qualified based on professional judgment.

The quantity of tracer material used should be adequate to provide a maximum of 10% uncertainty at the 95% confidence level in the measured recovery.

Evaluation	Action
If a recovery for a chemical carrier or tracer isotope is... >105% but ≤125%,	qualify all associated results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."
If a recovery for a chemical carrier or tracer isotope is... >125%,	qualify all associated results as "R."

Evaluation (concluded)	Action (concluded)
If a recovery for a chemical carrier is...	
<50% but ≥20%,	qualify all associated results ≥ the MDA as "J+."
<20%,	qualify all associated results ≥ the MDA as "J+" and all associated results < the MDA as "R."
If a recovery for a tracer isotope is...	
≥10% but <50%,	qualify all associated results ≥ the MDA as "J+."
<10%,	qualify all associated results ≥ the MDA as "J+" and all associated results < the MDA as "R."

4.7.4 MS

MS analyses are performed on field samples, except as noted below, as a measure of the ability to recover the analyte from a particular matrix.

Criteria: The MS data shall not be used to evaluate sample data unless the MS sample was from the same client and of similar matrix.

The recovery acceptance criteria for MS results must be within 75% to 125% unless the sample result is >4X the spike added (see Section 4.1.20).

One MS sample shall be analyzed from each batch, with a minimum frequency of one per 20 samples.

Samples identified as FBs or EBs shall not be used to satisfy the spike analysis requirement.

If an MS result fails to meet recovery criteria, all associated samples shall be redigested and reanalyzed. Unfiltered water samples and unprepared solid samples are exempt from the reanalysis requirement. Results for unfiltered water samples and unprepared solid samples for which the MS failed the acceptance criteria may be reported and qualified without reanalysis.

MSs are not required for gamma spectroscopy, radon-222, or any analyses utilizing standard addition spike or a tracer or carrier that is chemically identical to the analyte. In addition, radium-226 analyses that employ a barium-133 tracer

are exempt from the MS requirements. For radium-228 analysis, an MS is required if the final actinium separation, which is not traced by barium-133, does not incorporate a carrier recovery.

Evaluation	**Action**
If the MS sample was from another client or of a dissimilar matrix, the frequency criteria of the MS was not met, no MS was analyzed, or an FB or EB was used for the MS,	qualify all results ≥ the MDA as "J." The program may require results < the MDA to be qualified "UJ."
If an MS %R is…	
<25%,	qualify all associated results ≥ the MDA as "J-" and all associated results < the MDA as "R."
<75% but ≥25%,	qualify all associated results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."
>125% but ≤150%,	qualify all associated results ≥ the MDA as "J+."
>150%,	qualify all associated results as "R."

4.7.5 Replicate

Replicate analyses indicate laboratory precision based on each sample matrix.

If an MS/MSD was analyzed in place of a replicate, the following criteria are applied to the MS/MSD results. If insufficient sample was submitted to analyze a replicate or MS/MSD, the laboratory may analyze an LCS/LCSD to measure precision using the following criteria for evaluation.

Criteria: One replicate sample shall be analyzed from each batch with a minimum frequency of one per 20 samples. The replicate data shall not be used to evaluate associated sample data unless the replicate sample was from the same client and of similar matrix.

The RER calculated using the 2-σ TPU is used to determine replicate precision for radiochemical results.

The radiochemical replicate determinations shall agree when the 95% confidence level uncertainties are considered. That is, the RER shall be <1.0.

Samples identified as FBs or EBs shall not be used to satisfy the replicate analysis requirement.

No precision criteria applies to samples with activities < the MDA, including those where one result is > the MDA and one result is < the MDA.

Replicate analyses may not be possible in tritium analyses when the moisture content is too low or the sample size is too small. A discussion of this problem shall be included in the laboratory case narrative, with no qualifiers applied.

Evaluation	**Action**
If no replicate sample, no MSD, and no LCSD was analyzed for each matrix or for each data package,	qualify all results ≥ the MDA of the same matrix as "J." The program may require results < the MDA to be qualified "UJ."
If frequency criteria are not met, or if an FB or EB was used for the replicate,	qualify all results ≥ the MDA as "J." The program may require results < the MDA to be qualified "UJ." **Note**: Some programs may not require replicate evaluation on non-client samples. For these programs, note this in the data validation report, with no qualifications applied.
If the RER is >1.0 and ≤3.0,	qualify all associated results ≥ the MDA as "J." The program may require results < the MDA to be qualified "UJ."
If the RER is >3.0,	qualify all associated results for that analyte as "R." **Note:** Tritium in soils are not qualified "R" when the RER is >3.0.

4.7.6 LCS

The LCS serves as a measure of the overall performance of all steps in the analysis, including sample preparation.

Criteria: One LCS shall be analyzed for each batch up to 20 samples.

For aqueous LCS analytical results, the recovery acceptance criteria shall be within 80% to 120% of the true value. For solid LCS results, the recovery acceptance criteria shall be within the control limits specified by the agency

that prepared the reference material or statistically-derived limits developed by the laboratory. The laboratory shall report the control limits in the QC portion of the deliverable. Multiple LCS analyses may not be used to meet acceptance criteria; that is, if multiple LCSs are analyzed for a batch and any failures occur, the failed LCS will be used to qualify the data.

Evaluation	**Action**
If LCS frequency criteria are not met,	note the deficiency in the data validation report.
If the LCS %R for any analyte is…	
<30% or >150%,	qualify all associated sample results as "R."
< the lower control limit but ≥30%,	qualify all associated sample results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."
> the upper control limit but ≤150%,	qualify all associated sample results ≥ the MDA as "J+."
If an LCS/LCSD pair was analyzed and recoveries of any target analyte were both above and below acceptance criteria,	qualify all results ≥ the MDA as "J."
If an aqueous LCS was used for solid matrices,	qualify all associated results≥ the MDA as "J." The program may require results < the MDA to be qualified "UJ."

4.7.7 Instrument Control Charts

In general, there are four types of control charts used to monitor radiochemistry instrumentation performance: efficiency, resolution, centroid, and background.

Efficiency Control Charts: Used for all instrumentation. A radioactive control source (that does not have to match the counting geometry of the samples) is counted and decay-corrected counts, count rate, activity or efficiency of the source is plotted. Displayed on the same plot are the average, ±2-σ control limits, and ±3-σ control limits, or just simply upper and lower control limits. Since the frequency of instrument calibration typically ranges from monthly to annually, a control

source is counted to show that the instrument response is stable and that the efficiency calibration is valid for the sample count.

Resolution Control Charts: Used for instrumentation that utilize multi-channel analyzers to create spectra (with the exception of liquid scintillation counters). The full-width half-maximum (FWHM) of one or multiple peaks of the control source are plotted. Displayed on the same plot are the upper and lower FWHM control limits. This plot shows that there is no increase in instrument noise to negatively impact spectral resolution for the sample count.

Centroid Control Charts: Used for instrumentation that use multi-channel analyzers to create spectra (with the exception of liquid scintillation counters). The centroid of one or multiple peaks of the control source is plotted. Displayed on the same plot are the upper and lower centroid control limits. This plot shows that the instrument gain is stable and that drift that could lead to poor peak integration or misidentification of peaks has not occurred.

Background Control Charts: Used for most instrumentation, though has limited value for data validation. An instrument background is performed and the counts or count rate is plotted. Displayed on the same plot are the average, ±2-σ limit, and ±3-σ control limits, or just simply upper and lower control limits. This plot shows that the detector has not become contaminated with radioactivity or that the instrument noise has not increased to the point to cause unwanted counts.

For evaluation, a data point outside the control limit means outside the ±3-σ control limit when the laboratory provides σ-type control charts.

Instead of control charts, the laboratory may provide control summaries that provide control data of multiple detectors and/or types of charts. This is acceptable as long as all the information needed to evaluate instrument control is provided in the summary.

4.7.7.1 All Radiochemistry Instrumentation

Criteria: The instrument raw data will clearly contain the detector ID and count start date and time for all samples. The control charts will list the detector IDs and list the range of dates plotted. The date range must be current up to the count start date of the sample. For controls that are counted daily or before use, the charts must be updated to the actual date of sample count. For controls that are counted weekly/monthly, the charts must be updated to within a week/month of the sample count. See the specific instrumentation criteria below for control count frequency requirements.

In general, only the control chart data point appropriate for the sample count start date is evaluated. If the next control chart data point is plotted and shows an extreme outlier, the stability of the counter during the time of the sample count may need to be investigated.

The laboratory should have made an attempt to recount the sample (if possible) and verify the original count results. Professional judgment is needed in this situation, especially if the sample result looks suspect.

Evaluation	Action
If the control chart is missing from the data package or not updated,	request an amended report from the laboratory.
If instrument control frequency was not met for the sample count,	note the deficiency in the data validation report.

4.7.7.2 Gas Proportional Instrumentation

Criteria: Alpha and beta control sources will be counted daily or before counter use and plotted using efficiency control charts. A background will be counted daily or before counter use and plotted using alpha and beta background control charts.

If the sample analyte is an alpha-emitter, only the alpha control charts are evaluated. If the sample analyte is a beta-emitter, only the beta control charts are evaluated.

Evaluation	Action
If the efficiency control point is…	
above the upper control limit,	qualify all associated results ≥ the MDA as "J+"
below the lower control limit,	qualify all associated results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."
If the background control point is…	
above the upper control limit,	qualify all associated results ≥ the MDA but <3X the MDA as "J+."
below the lower control limit,	qualify all associated results ≥ the MDA but <3X the MDA as "J-." The program may require results < the MDA to be qualified "UJ."

4.7.7.3 Liquid Scintillation Instrumentation

Criteria: Vendor supplied unquenched H-3, C-14, and blank control sources will be counted daily or before counter use and plotted using efficiency and background control charts.

For low-energy beta analysis, (such as H-3, Ni-63 or Pu-241) only the H-3 efficiency and background control charts are evaluated. For mid- to high-energy beta analysis, (such as C-14, Cl-36, Sr-90 or Tc-99) only the C-14 efficiency and background control charts are evaluated.

Evaluation	**Action**
If the efficiency control point is…	
above the upper control limit,	qualify all associated results ≥ the MDA as "J+."
below the lower control limit,	qualify all associated results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."
If the background control point is…	
above the upper control limit,	qualify all associated results ≥ the MDA but < 3X the MDA as "J+."
below the lower control limit,	qualify all associated results ≥ the MDA but <3X the MDA as "J-." The program may require results < the MDA to be qualified "UJ."

4.7.7.4 Lucas Cell Instrumentation

Criteria: A control source will be counted daily or before counter use and plotted using efficiency control charts.

Evaluation	**Action**
If the efficiency control point is…	
above the upper control limit,	qualify all associated results ≥ the MDA as "J+."
below the lower control limit,	qualify all associated results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."

4.7.7.5 Alpha Spectrometer Instrumentation

Criteria: If calibrated monthly, the calibration standard can also be used as the control source for generating efficiency control chart data. If a semi-annual or annual calibration is performed, a control source will be counted at least monthly and plotted using efficiency control charts. Only one peak is necessary to control chart. The laboratory should perform pulser control checks at least weekly (preferably daily or before use). The pulser checks will confirm that the instrument gain and resolution are stable. Backgrounds will be counted at least weekly and monitored by the laboratory for contamination. Pulser check results and background control charts do not have to be included or evaluated in the data package.

Evaluation	Action
If the efficiency control point is outside of the control limits and the tracer is measured simultaneously with the analyte,	note the deficiency in the data validation report. **Note:** Although the analyte results will not be biased, the reported tracer yield may be biased.
If an efficiency control point is above the upper control limit and a tracer is not measured simultaneously with the analyte (Ba-133, Np-239 or Th-234),	qualify all associated results ≥ the MDA as "J+."
If an efficiency control point is below the lower control limit and a tracer is not measured simultaneously with the analyte (barium-133, neptunium-239, or thorium-234),	qualify all associated results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."
If the detector background was not counted within one week of the sample count start date,	note the deficiency in the data validation report.

4.7.7.6 Gamma Spectroscopy Instrumentation

Criteria: A source will be counted daily or before counter use and plotted using efficiency control charts. At a minimum, two peaks need to be control charted for efficiency, resolution (FWHM), and centroid. These peaks are a low-energy peak (< 100 kilo electron volts [kev]) and a high-energy peak (> 1000 kev). Backgrounds will be counted at least weekly and monitored by the

laboratory for contamination; however, background control charts do not have to be included or evaluated in the data package.

If the low-energy efficiency control point is outside control limits and the high-energy control point is within limits, technically only the low-energy gamma emitting target analytes need qualification. Since the determination of what energy range requires qualification is not straightforward and requires professional judgment, it is acceptable to qualify all target analytes in this situation.

Evaluation	Action
If the efficiency control point is…	
above the upper control limit,	qualify all associated results≥ the MDA as "J+."
below the lower control limit,	qualify all associated results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."
If the resolution control point is outside (above or below) of the control limits,	notify the laboratory and qualify all associated sample results as "R."
If the centroid control point is outside (above or below) of the control limits,	note the deficiency in the data validation report.
If the detector background was not counted within one week of the sample count start date,	note the deficiency in the data validation report.

4.7.8 Method-Specific Analytical Requirements – Radiochemical

4.7.8.1 Gamma Spectroscopy

The laboratory may rejection of a specific gamma spectroscopy analyte result due to various analytical quality issues (e.g., interference, low abundance, no valid peak, or uncertain identification). This data shall be assessed based on professional judgment.

Criteria: The laboratory qualifiers shall be reviewed for "X"- qualified data.

Evaluation	Action
If the result is recommended for rejection by the laboratory,	may qualify the result as "R" based on professional judgment.

4.7.8.2 Gross Alpha Beta

The flaming of planchets in the gross alpha beta method may result in the loss of beta emitters. The omission of the flaming step may result in the interference of alpha particle transmission.

Criteria: The sample preparation documentation shall be examined to determine whether the planchets were flamed.

Evaluation	Action
If the planchets were flamed prior to counting for gross beta,	qualify all beta results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."
If the planchets were not flamed,	qualify all alpha results ≥ the MDA as "J-." The program may require results < the MDA to be qualified "UJ."

4.7.8.3 Total/Partial Radiochemical Results

Occasionally radiochemical analytes are analyzed and reported as total and partial results, for example, total radium by gross alpha and radium-226 by radon emanation. These reported values are necessarily not obtained from the same analytical method. The following criteria should be used for guidance.

Criteria: When both a partial and a total result are reported, the result for the partial analyte must be ≤ the result for the total analyte. If the reported result for the partial analyte is > the result for the total analyte, one or both results are suspect.

The extent the quality of the data is affected must be determined.

Evaluation	Action
If the partial result is > the total result,	the laboratory should be contacted for further information.
If the laboratory cannot be contacted or cannot provide sufficient explanation, the following criteria apply: If the total result is ≤ the MDA and the partial result is > MDA but < 2X MDA,	no qualification (other than "BD" or "J" due to quantification) of either result is warranted as the results are statistically similar enough.

Evaluation (concluded)	Action (concluded)
If the total result is ≤ the MDA and the partial result is > 2X MDA,	qualify the total result as "J-."
If both results are > MDA and the total result is < the partial, and the RER between the two results is >1.0,	qualify the total result as "NJ-" due to a suspected false negative.

5.0 DATA VALIDATION REPORTS

A data validation report shall be produced to discuss the data review and validation; and to document, based on instrumentation and methodology, the QC elements examined. The SMO or Program Manager uses the data validation report to evaluate and determine if nonconformance, corrective actions, or penalties should be pursued. If this procedure is modified based on the professional judgment of the data validator, the data validation report must document the adjustments. Any method-specific QC requirements not addressed in this document must be documented by the data validator in the data validation report or included as an addendum to the procedure. The database administrator submits the data validation report to the SNL/NM Customer Funded Record Center for archiving. The data validation report shall include the following (as appropriate):

5.1 Sample Findings Summary and Validation EDD Files

A data table or spreadsheet summarizing flagged data resulting from the data review and validation. The sample findings summary is to be used by database personnel to facilitate the data entry of data validation qualifiers to the electronic database. However, when laboratory EDD files are available the SNL/NM Validation EDD Generator is used to produce a sample findings summary and validation EDD file. The validation EDD file is subsequently used for electronic data entry of data validation qualifiers to the database. The sample findings summary and validation EDD file shall include the following:

- The site name
- ARCOC number
- Sample number(s)
- Analysis or individual analytes
- Data validation qualifiers
- Any relevant comments

5.2 Data Validation Narrative (format may vary by project)

A summary of samples and all qualifiers applied to the data as a result of the validation process. The narrative shall include the following:

- The date issued
- The names of those to whom the report is issued
- The validator's name
- The laboratory name and SDG identifier
- ARCOC number
- Type of analysis addressed in the report
- Sample/analyte qualification and a general description of why qualification was applied
- Data validation procedure and revision used
- Any relevant comments

5.3 Data Qualification Summary

A summary of the process used for review and validation. The data qualification summary includes the following sections **(as appropriate)**:

- Sample Shipping/Receiving (refer to Section 4.1.1). Are all shipping/receiving and ARCOC issues that could affect data quality and defensibility discussed, and qualifications properly applied?
- Holding Times and Preservation (refer to Section 4.1.1). Are all holding time and preservation issues that could affect data quality discussed, and qualifications properly applied?
- Calibration. Are all calibration (initial and/or continuing/verification) issues that could affect data quality discussed, and qualifications properly applied?
- Tuning. Are all tuning issues that could affect data quality discussed, and qualifications properly applied?
- IS. Are all IS issues that could affect data quality discussed, and qualifications properly applied?
- Isotope Ratios. Are all isotope abundance issues that could affect data quality discussed, and qualifications properly applied?
- Surrogates. Are all surrogate issues that could affect data quality discussed, and qualifications properly applied?
- TICs. If required, are the identification and qualification of TICs discussed?
- Confirmation. Was second-column analysis discussed, and were qualifications properly applied?
- RLV (CRI/CRA/CRDL). Are all RLV and CRI/CRA/CRDL issues that could affect data quality discussed, and qualifications properly applied?
- ICP ICS. Are all ICS issues that could affect data quality discussed, and qualifications properly applied?
- ICP SD. Are all SD issues that could affect data quality discussed, and qualifications properly applied?
- Tracer/Carrier. Are all tracer and/or carrier issues that could affect data quality discussed, and qualifications properly applied?
- Blanks. Are all detections of target analytes in all applicable blanks discussed, and qualifications properly applied?

- LCS. Are all LCS issues that could affect data quality discussed, and qualifications properly applied?
- MS. Are all MS issues that could affect data quality discussed, and qualifications properly applied?
- Laboratory Replicates. Are all the laboratory replicate issues that could affect data quality discussed, and qualifications properly applied?
- DLs/Dilutions. Is the appropriateness of the reported DLs discussed? Are sample dilutions discussed?
- Other QC. Are all QC issues that could affect data quality, other than those previously addressed, discussed? Include a brief description of any laboratory nonconformance reports that directly impacted data quality.
- Corrective Action Reports. Discuss or attach laboratory correspondence covering any corrective action, clarification, or modification to the report that was required to complete the validation process.

5.4 Validation Notes/Worksheets (as appropriate)

The validation notes/worksheets document results of the review and data validation by methodology and show QC results that do not meet acceptance criteria (that is, failures). These notes/worksheets identify data for which holding time/preservation requirements and calibration acceptance criteria were not met; laboratory blanks, FBs, EBs, or TBs were contaminated; surrogate recovery criteria were exceeded; MS/MSDs exceeded limits; and LCS %Rs and replicate RPDs or RERs exceeded acceptance limits. In addition, the validation notes/worksheet identify the validator and the laboratory; include the validator's comments and notes; and include ARCOC numbers, SDG number, types and number of samples analyzed, and sample numbers.

5.5 CVR and ARCOC

The CVR and ARCOC records also are pertinent to data review and validation. These records are supplied by the SMO and the laboratory and are copied and attached to the data validation report.

6.0 DEFINITIONS

6.1 Data Qualifier Definitions

Data qualifiers are commonly used during the validation process to classify sample data as to their conformance to QC requirements. For the purposes of this procedure, the following code letters and associated definitions are provided:

BD	(below DL) - Used in radiochemistry to identify results that are not statistically different from zero.
J	The associated value is an estimated quantity.
J+	The associated numerical value is an estimated quantity with a suspected positive bias.
J-	The associated numerical value is an estimated quantity with a suspected negative bias.
N	Presumptive evidence of the presence of the material.

NJ Presumptive evidence of the presence of the material at an estimated quantity.

NJ+ Presumptive evidence of the presence of the material at an estimated quantity with a suspected positive bias.

NJ- Presumptive evidence of the presence of the material at an estimated quantity with a suspected negative bias.

R The data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.

U The analyte was analyzed for but was not detected. The associated numerical value is the sample quantitation limit.

UJ The analyte was analyzed for but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

Datum is unqualified if the quality parameters indicate the method was appropriate and that the reported result reflects the true value within the expected analytical uncertainty.

Datum is qualified as estimated (J) if the reported result can be used to infer an estimate of the true value (with a suspected positive or negative bias, as may be indicated), but the quality parameters indicate an uncertainty in the result that is > the expected analytical uncertainty.

Datum is qualified as presumptive (N) if there is question as to whether the analyte is indigenous to the sample or if there is question regarding the identity of the analyte.

Datum is qualified as presumptive and estimated (NJ) when there is evidence of the presence of the material at an estimated quantity (with a suspected positive or negative bias, as may be indicated).

Datum is qualified as unusable (R) if the quality parameters do not support the reported result as a valid indicator of the true value.

Datum is qualified as estimated non-detects (UJ) for results reported as < the DL and for which some other quality concerns exist.

6.2 Sample Quantification Limits

For purposes of this procedure, the following definitions are provided:

MDA Minimum detectable activity. A radiological DL. A sample with activity concentration at the minimum detectable concentration (MDC) has a 95% probability of being measured above the decision level, which is the lowest threshold used to distinguish a positive result (i.e., a detect). For the purposes of data validation, the MDC equals the minimum detectable activity.

MDL Method detection limit. The minimum concentration of a substance that can be measured (quantified) and reported with 99% confidence that the analyte concentration is > zero. This measure of instrument sensitivity takes into account all solutions that have been subjected to all sample preparation steps for the method. In data packages the MDL may be referred to as the DL. For organic data, the MDL will be one-fifth the PQL, and the

value associated with the "U" qualifier or the value of the low standard will be used as the PQL.

PQL Practical quantitation limit. The lowest concentration of analytes in a sample that can be reliably determined and quantified within specified limits of precision and accuracy by the indicated methods under routine laboratory operating conditions. For the purposes of this procedure, the PQL is considered to be 5X the value of the MDL if not defined by the laboratory. In data packages the PQL may be referred to as the contract-required DL or RL. For inorganic data, the PQL will be 5X the MDL, and the value associated with the "U" qualifier will be used as the MDL.

6.3 Formulas

The %D for LCSs, standards, and SD is calculated as follows:

$$\%D = \frac{[M - T]}{T} \times 100$$

where,
- %D = percent difference
- M = measured value
- T = true value or sample value for SD

The RPD for replicate samples is calculated as follows:

$$RPD = \frac{[S - R]}{(S + R)/2} \times 100$$

where,
- RPD = relative percent difference
- S = sample value (original)
- R = replicate sample value

The RPD for MS/MSD samples is calculated as follows:

$$RPD = \frac{MS - MSD}{(MS+MSD)/2} \times 100$$

where,
- RPD = relative percent difference
- MS = MS value
- R = MSD value

The %R for spiked samples is calculated as follows:

$$\%R = \frac{SSR-SR}{SA} \times 100$$

where, SSR = spiked sample result
SR = sample result
SA = spike added

The RER is used to determine replicate precision for radiochemical results. The RER is given by:

$$RER = \frac{[S - R]}{F_{95S} + F_{95R}}$$

where, RER = replicate error ratio
S = sample value (original)
R = replicate sample value
F_{95S} = sample uncertainty (95% or 2-σ)
F_{95R} = replicate uncertainty (95% or 2-σ)

The linear curve equation is given by:

$$y = mx + b$$

where, y = instrument response (peak area or height)
m = slope of the line (also called the coefficient of x)
x = concentration of the calibration standard
b = the intercept

The %RSD is calculated as follows:

$$\%RSD = \frac{SD}{RF} \times 100\%$$

where, SD = standard deviation
RF = mean RF for each compound from the ICAL

7.0 GLOSSARY OF TERMS

2-σ error: The error reported at the 95% confidence interval.

Acceptance limits: Ranges of acceptable results for each type of QC measurement. They may be defined on a program-specific basis, or they may be derived internally at a laboratory from historic QC performance data. May also be referred to as control limits.

Accuracy: The closeness of agreement between an observed value and the true value. "Precision" is a measure of the reproducibility of a value, without knowledge of the true value. The classic example used to illustrate these terms is a dartboard example: The placement of four darts thrown at a dartboard is considered accurate if the darts are each close to the bull's-eye (regardless of their proximity to one

another). Hence, to be both accurate and precise the four darts would need to be grouped closely together and be close to the bull's-eye.

Analyte: That which is analyzed for. This can be chemical (chromium, benzene), biological (fecal coliform bacteria), mineral (asbestos fibers), or radiological (alpha and beta emissions).

Analytical run: The interval (i.e., period of time or series of measurements) within which the accuracy and precision of the measuring system is expected to be stable. Within the analytical run, controls are often analyzed to confirm stability.

Batch: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is > 20, then each group of 20 samples or less will all be handled as a separate batch.

Bias: The difference between the reported result and the true result. Bias may be introduced through field or laboratory variability and error or due to substances in the sample that interfere with the analytical system's ability to provide an accurate measurement. Because the true concentration of an analyte in an environmental sample is generally never known, bias is estimated by using surrogates, MSs, LCSs, and other indicators of analytical accuracy.

Calibration: The process of correlating instrument signal response with analyte concentration. An instrument must be properly calibrated in order to produce accurate results.

Chemical carrier: An identical or similar carrier material used to infer the degree to which the separation processes were effective in separating the analyte from the matrix. Measured gravimetrically or chemically.

Congener: A congener refers to any one particular compound of the same chemical family. For example, there are 209 congeners of Chlorinated Biphenyls (CBs).

Contamination: A component of a sample from an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments. Blanks (instrument blanks, MBs, preparation blanks, TBs, EBs, and FBs) may be used to assess contamination.

Control sample: A QC sample introduced into a process to monitor the performance of the system.

Correlation coefficient (r) or coefficient of determination (r^2): A statistical evaluation of the linearity of a calibration curve, i.e. "goodness of fit."

Detect: Sample result ≥the MDL.

Duplicate: A second aliquot of a sample that is treated the same as the original aliquot of sample. (See definitions for field duplicate and replicate.)

Environmental sample: A sample taken unaltered (as much as possible) from the environment (as opposed to a blank, performance evaluation sample, MS sample, etc.). Environmental sample may be referred to as "field sample."

Equipment blank (EB): A sample of analyte-free media (for example, clean water poured over a bailer) that has been used to rinse the sampling equipment. The EB is collected after completion of decontamination and prior to collection of environmental samples. This blank is useful in documenting adequate decontamination of sampling equipment. An EB also may be referred to as a "rinsate blank."

Field blank (FB): A sample containing an analyte-free matrix that is collected and processed in exactly the same manner as an equivalent environmental sample (for example, clean water is poured into a sample container in the same physical location where the environmental sample is collected and is subsequently handled, processed, and analyzed exactly as an equivalent environmental sample). The FB is used to identify contamination resulting from field sample collection techniques.

Field duplicate: A duplicate sample generated in the field used to determine sampling and analytical precision.

Holding time: The period between collection of samples by the samplers and preparation and/or analysis of samples by the laboratory (see Appendix B for required hold times). If the method specifies a holding time to extraction and a holding time to analysis then two holding times are evaluated. If no holding time to extraction is specified then the listed holding time is the holding time to analysis. That is, the laboratory cannot extract a sample and store the extract in order to meet holding time. However, professional judgment may be applied here. If the sample preparation includes MS and LCS samples and both of these pass it may be inferred that the stability of the extract has been verified.

Instrument blank: A blank designed to determine the level of contamination associated with the analytical instruments.

Internal standard (IS): A chemical compound added to every blank, sample, and standard extract at a known concentration that is used to (1) compensate for analyte concentration changes that might occur during storage of the extract, and (2) compensate for quantification variations that can occur during analysis. ISs are used as the basis for quantifying target analytes.

Isomer: A chemical species with the same number and types of atoms as another chemical species, but possessing different properties. For example, 2,3,7,8-TCDD refers to only one of the 22 possible TCDD isomers; that isomer which is chlorinated in the 2,3,7,8-position of the dibenzo-p-dioxin ring structure.

Isotope dilution: A means of determining a naturally occurring (native) compound by reference to the same compound in which one or more atoms has been isotopically enriched.

Laboratory control sample (LCS): A known matrix that is spiked with compounds representative of the target analytes at known concentrations. The spiking occurs prior to sample preparation and analysis. An LCS is used to document laboratory overall performance.

Matrix: The substrate that contains the analyte of interest (for example, surface water, drinking water, air, soil, tissue, etc.).

Matrix interference: Bias introduced because something in the sample interferes with the analytical system's ability to provide an accurate measurement. The interference may be physical (turbidity in stormwater runoff may block light transmission in an analysis based on ultraviolet absorbance) or chemical (a chemical similar to the analyte of interest may increase the response of the instrument, resulting in a positive bias).

Matrix spike (MS): A measured amount of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. An MS is used to assess the bias of a method in a given sample matrix.

Matrix spike duplicate (MSD): Intralaboratory (within the same laboratory) split-samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. MSDs are used to assess the precision and bias of a method in a given sample matrix.

Method blank (MB): An analyte-free matrix that is prepared and processed at the laboratory in exactly the same manner as an equivalent environmental sample (that is, all reagents are added in the same volumes or proportions as used in sample processing). The MB is used to document contamination resulting from the analytical process.

Non-detect: Sample result < the MDL.

Ongoing Precision and Recovery (OPR): A MB spiked with known quantities of analytes and analyzed as a sample. Its purpose is to assure that results produced by the laboratory remain within the limits specified in EPA Method 1668A for precision and recovery.

Precision: The proximity to one another of the results for multiple measurements on the same sample (i.e., a measure of the repeatability of a measurement process). This does not address proximity to a true value; it is possible for multiple results to show very high precision and yet be completely incorrect by comparison with a true value. Precision is quantified, for example, by calculating the RPD between the result obtained for a sample and that obtained from an associated duplicate or replicate sample. As with accuracy, there is an assumed correlation between quantitative precision as determined via QC analyses and the inferred precision in measurements of unknowns.

Quality control (QC): The system of routine technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. In other words, QC activities are the tactics used to measure and control quality.

Radioactive tracer: A radioactive isotope of the analyte that is added to the sample to correct for any losses of the analyte during the chemical separations or other processes employed in the analysis.

Relative dilution factor: The dilution factor ratio (value ≥1) of two samples. For example, if one sample has a dilution factor of 2 and another sample has a dilution factor of 10, the relative dilution factor is 5.

Relative response factor (RRF): A measure of the relative mass spectral response of an analyte compared to its IS. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

Relative retention time (RRT): The ratio of the RT of a compound to that of a standard (such as an IS).

Replicate (also may be called "sample duplicate"): A duplicate sample generated in the laboratory used to determine analytical precision.

Reporting limit verification (RLV): A low-level verification standard of the same origin as the calibration standard run as a measure of accuracy near the PQL. The RLV is known as the CRI for ICP-AES, ICP-MS, and LC/MS/MS methods, CRA for AA methods, and CRDL for cyanide methods.

Response (also may be called "instrument response"): The signal output of an analytical instrument in which the intensity of the signal is proportionate to the concentration detected. Response is measured by peak area or peak height.

Retention time (RT): The time a target analyte is retained on a chromatography column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT window established for that compound. RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

Sample delivery group (SDG): A group of samples that are processed together by the laboratory. Ideally, all the samples in a batch will be similar enough that matrix QC measurements performed with the batch will be representative of all the samples in the batch.

Spike: A known amount of analyte that is introduced purposely into a sample (either an environmental sample or a blank) for the purpose of determining whether the analytical system can accurately measure the analyte.

Surrogate: A chemical that is similar to the target analyte(s) in chemical composition and behavior in the analytical process but that is not expected to be present in the sample. Surrogates are added to all the environmental samples, blanks, and QC samples in the analytical batch during the preparation stage of the analysis. Surrogates are used to monitor the performance of the analytical process. An example would be the use of fluorinated organic compounds in an analysis that looks for chlorinated and brominated organic compounds. Surrogates also may be called "system monitoring compounds" (SMCs).

Target analyte: A chemical that is being looked for in an analysis.

Tentatively identified compound (TIC): A compound that is outside the standard list of analytes in a GC/MS method but that is reported based on a tentative match between the instrument response and the instrument's computer library. The identification and quantitation of these compounds is tentative.

Trip blank (TB): A sample of analyte-free media (such as distilled/deionized water) taken from the laboratory to the sampling site and returned to the laboratory unopened. A TB is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organic samples.

8.0 REASON CODES

Programs may require that general validation codes be included in their computer databases or EDDs. The following codes are the default codes used when this is required.

H1 - Holding time exceeded for sample analysis
H2 - Holding time exceeded for sample extraction
H3 - Holding time exceeded by >2X the specified holding time

TP1 - Sample improperly preserved
TP2 - Sample not preserved
TP3 - Sample not maintained at required temperature
TP4 - Required sample or extract clean up not performed
TP5 - Sample received unpreserved and preserved at the laboratory

I1 - Initial calibration not reported
I2 - Initial calibration not independently verified
I3 - Slope r^2 or RF %RSD criteria not met
I4 - Minimum RF / Slope not met
I5 - Intercept too large
I6 - Insufficient number of calibration standards used

C1 - Continuing calibration frequency not met
C2 - Continuing calibration %D failed high
C3 - Continuing calibration %D failed low

B - MB contamination at concentration >MDL
B1 - TB contamination at concentration >MDL
B2 - FB/EB contamination at concentration >MDL
B3 - Calibration blank contamination at concentration >MDL
B4 - Negative value for calibration blank - absolute value >the MDL
B5 - Negative value for MB - absolute value >the MDL
B6 - Negative value for FB/EB/TB - absolute value >the MDL
B7 - MB contamination at activity ≥the MDA
B8 - MB frequency not met
B9 - Instrument or calibration blank frequency not met

IS1 - IS / tracer recovery failed high
IS2 - IS / tracer recovery failed low but ≥10%
IS3 - IS / tracer recovery failed low and <10%

S1 - Surrogate(s) failed high
S2 - Surrogate(s) failed low
S3 - Multiple random surrogate failures

FR1 - Result exceeds calibration range
FR2 - No result reported - sample lost or damaged
FR3 - Result is less than the MDA / MDL or < the 2-σ TPU
FR4 - Negative result - absolute value >2X the MDA/MDL

FR5 - RT criteria not met
FR6 - Ion mass ratio criteria not met
FR7 – Result is ≥ the MDA and <3X the MDA

MS1 - MS not analyzed or not applicable
MS2 - MS analyte(s) recovery failed high
MS3 - MS analyte(s) recovery failed low
MS4 - MS analytes recovery failed both high and low
MS5 - MS/MSD RPD failed

RP1 - Replicate not analyzed or not applicable
RP2 - Replicate RPD failed

L1 - LCS frequency not met
L2 - LCS analyte(s) recovery failed high
L3 - LCS analyte(s) recovery failed low
L4 - LCS analytes recovery failed both high and low
L5 - LCS/LCSD RPD failed

DL1 - RLV frequency not met[1]
DL2 - RLV percent recovery failed high[1]
DL3 - RLV percent recovery failed low[1]

CK1 - ICS frequency not met
CK2 - ICS analyte(s) failed high
CK3 - ICS analyte(s) failed low

D1 - SD failed %D
D2 - Inappropriate initial dilution

V1 - Conformation analysis not done
V2 - Conformation RPD exceeds criteria
V3 - Confirmation analysis by second method did not confirm original result (LCMSMS)

X1 - Non-specified data quality concern – see validation report
X2 - Analysis failed to meet method requirements - see validation report
X3 - Required QC documentation missing

Z1 - Spectral identification criteria not met[2]
Z2 - Minimum peak criteria not met[3]

1: refers to QC for CRA/CRDL/CRI analyses.
2: used when rejecting results that have been X qualified by the lab for interference or short half-life, and also for failed organic spectral matching (GC/MS, diode array, HPLC, etc.).
3: used when rejecting results that have been X qualified by the lab for low abundance, no valid peak, or peak not meeting identification criteria.

9.0 REFERENCES

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SNL/NM Statement of Work for Analytical Laboratories, Current Revision, Sample Management Office, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

Model Statement of Work for Analytical Laboratories (prepared for the DOE NNSA Service Center)

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USEPA Contract Laboratory Program National Functional Guidelines

40 CFR 136, Protection of Environment: Guidelines Establishing Test Procedures for the Analysis of Pollutants

EPA Method 314, *Determination of Perchlorate in Drinking Water by Ion Chromatography*

EPA Method 1613B, *Tetra-thru-Octa (CDDs) Chlorinated Dioxins and Furans (CDFs)*

EPA Method 1668A, *Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)*

EPA Method 1694, *Pharmaceuticals and Personal Care Products in Water, Soil, Sediment, and Biosolids by HPLC/MS/MS*

EPA Method TO-14A (Rev. 1), *Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters with Subsequent Analysis by Gas Chromatography*

EPA Method TO-15 (Rev. 1), *Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)*

SW-846 Method 428, *Determination of Polychlorinated Dibenzo-p-dioxin (PCDD), Polychlorinated dibenzofuran (PCDF), and Polychlorinated Biphenyl Emissions from Stationary Sources*

SW-846 Method 8000C, *Determinative Chromatographic Separations*

SW-846 Method 8015C, *Nonhalogenated Organics Using GC/FID*

SW-846 Method 8015D, *Nonhalogenated Organics Using GC/FID*

SW-846 Method 8081B, *Organochlorine Pesticides by Gas Chromatography*

SW-846 Method 8082A, *Polychlorinated Biphenyls (PCBs) by Gas Chromatography*

SW-846 Method 8151A, *Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization*

SW-846 Method 8260B, *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*

SW-846 Method 8260C, *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*

SW-846 Method 8270C, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*

SW-846 Method 8270D, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*

SW-846 Method 8280B, *The Analysis of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)*

SW-846 Method 8290A, *Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)*

SW-846 Method 8310, *Polynuclear Aromatic Hydrocarbons*

SW-846 Method 8330B, *Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)*

NIOSH 7300, Elements by ICP (Nitric/Perchloric Acid Ashing)

Appendix A

Volatile Organic Holding Times

Volatile Organic Compounds
Maximum Holding Times
Non-detects in Water

Compound	CAS #*	15 - 60 days	60 - 120 days
1,1-dichloroethene	75-35-4	UJ	R
2-butanone	78-93-3	UJ	R
Acrolein	107-02-8	UJ	R
Allyl chloride	107-05-1	UJ	R
Bromomethane	74-83-9	UJ	R
Carbon tetrachloride	56-23-5	UJ	R
Chloromethane	74-87-3	UJ	R
Dibromochloromethane	124-48-1	UJ	R
Ethyl benzene	100-41-4	UJ	R
m&p xylene	Na	UJ	R
Methyl methacrylate	80-62-6	UJ	R
o xylene	95-47-6	UJ	R
Vinyl chloride	75-01-4	UJ	R
1,1,1 trichloroethane	75-55-6	Not Qualified	UJ
1,1 dichloropropene	563-58-6	Not Qualified	UJ
1,2,4 trichlorobenzene	120-82-1	Not Qualified	UJ
1,2,4 trimethylbenzene	95-63-6	Not Qualified	UJ
1,2 dibromo-3-chloropropane	96-12-8	Not Qualified	UJ
1,2 dichlorobenzene	95-50-1	Not Qualified	UJ
1,3,5 trimethylbenzene	108-67-8	Not Qualified	UJ
1,3 dichlorobenzene	541-73-1	Not Qualified	UJ
1,4 dichlorobenzene	106-46-7	Not Qualified	UJ
4-methyl-2-pentanone	108-10-1	Not Qualified	UJ
Bromodichloromethane	75-27-4	Not Qualified	UJ
Chlorobenzene	108-90-7	Not Qualified	UJ
Chloroethane	75-00-3	Not Qualified	UJ
Ethyl methacrylate	97-63-2	Not Qualified	UJ
Hexachlorobutadiene	87-68-3	Not Qualified	UJ
Isopropylbenzene	98-82-8	Not Qualified	UJ
Naphthalene	91-20-3	Not Qualified	UJ
n-propylbenzene	103-65-1	Not Qualified	UJ
2-chlorotoluene	95-49-8	Not Qualified	UJ
4-chlorotoluene	106-43-4	Not Qualified	UJ
4-isopropyltoluene	99-87-6	Not Qualified	UJ
Tetrachloroethene	127-18-4	Not Qualified	UJ
Trichloroethene	79-01-6	Not Qualified	UJ

Volatile Organic Compounds
Maximum Holding Times
Non-detects in Water (concluded)

Compound	CAS #*	60-120 days	120 - 240 days
1,1,1,2 tetrachloroethane	630-20-6	Not Qualified	UJ
1,1,2,2 tetrachloroethane	79-34-5	Not Qualified	UJ
1,1,2 trichloroethane	79-00-5	Not Qualified	UJ
1,1 dichloroethane	75-35-4	Not Qualified	UJ
1,2,3 trichlorobenzene	87-61-6	Not Qualified	UJ
1,2 dibromomethane	106-93-4	Not Qualified	UJ
1,2 dichloroethane	107-06-2	Not Qualified	UJ
1,2 dichloropropane	78-87-5	Not Qualified	UJ
1,3 dichloropropane	142-28-9	Not Qualified	UJ
Acetone	67-64-1	Not Qualified	UJ
Acrylonitrile	75-05-8	Not Qualified	UJ
Benzene	71-43-2	Not Qualified	UJ
Bromobenzene	108-86-1	Not Qualified	UJ
Bromochloromethane	74-97-5	Not Qualified	UJ
Bromoform	75-25-2	Not Qualified	UJ
Chloroform	67-66-3	Not Qualified	UJ
cis-1,2 dichloroethene	156-59-2	Not Qualified	UJ
Dibromomethane	106-93-4	Not Qualified	UJ
Dichlorodifluoromethane	75-71-8	Not Qualified	UJ
Methyl acrylonitrile	126-98-7	Not Qualified	UJ
Methylene chloride	75-09-2	Not Qualified	UJ
n-butylbenzene	104-51-8	Not Qualified	UJ
sec-butyl benzene	135-98-8	Not Qualified	UJ
tert-butyl benzene	98-06-6	Not Qualified	UJ
Toluene	108-88-3	Not Qualified	UJ

*chemical abstract service number

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Appendix B

Sample Preservation and Holding Times

Sample Preservation Techniques and Holding Times

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times Extract
2310B, 2320B	Acidity, Alkalinity	Water	500 mL Plastic or Glass	≤6 °C	14 Days	NA
300.0, 300.1 375.2	Bromide, Chloride, Fluoride, Sulfate	Water	1 L Plastic	≤6 °C	28 Days	NA
5210B	BOD	Water	1 L Plastic	≤6 °C	48 Hours	NA
9010B, 9013, 9014, 335.4, 4500CN-G	Total Cyanide Amenable Cyanide	Water Solid/Other	1 L Plastic 125 mL Glass Jar	≤6°C; NaOH; pH > 12 ≤6°C	14 Days 14 Days	NA NA
5310B, C or D, 9060	DOC, TOC	Water	250 mL Amber Glass	≤6 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	≤6 °C	28 Days	NA
200.7, 200.8, 6010B, 6020A	All metals except Cr(VI) and Hg	Water Solid/Other	500 mL Plastic 250 mL Glass Jar	HNO_3 ; pH < 2	180 Days 180 Days	NA NA
3060A 218.6 7197, 7196A	Cr(VI)	Water Water Solid/Other	500 mL Plastic 500 mL Plastic 250 mL Glass Jar	≤6 °C ≤6°C, pH 9-9.5 ≤6 °C	24 Hours 28 Days 30 Days	NA NA 7 Days
245.1, 7470A, 7471A	Hg	Water Solid/Other	500 mL Plastic 250 mL Glass Jar	HNO_3 ; pH < 2 ≤6 °C	28 Days 28 Days	NA NA
130.1	Hardness	Water		HNO_3 ; pH < 2 ≤6 °C	180 Days	NA
345.1	Iodide	Water	500 mL Plastic or Glass	≤6 °C	24 Hours	NA

Note: 40 mL vials require caps with Teflon lined septum. All other containers require Teflon lined screw cap lids to minimize container contamination and loss of analyte.
*If samples are shipped to the laboratory in EnCore™ samplers, samples must be extruded and placed in sample containers within 48 hours of sample collection.

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Sample Preservation Techniques and Holding Times

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times Extract
353.2, 351.1	Ammonium, Nitrate + Nitrite,	Water	1 L Plastic	≤6 °C; H_2SO_4; pH < 2	28 Days	NA
351.2, 365.4	Total Phosphorus, TKN			≤6°C; not acidified	24 Hours	NA
350.1						
300.0	Nitrate, Nitrite,	Water	500 mL Plastic	≤6 °C	48 Hours	NA
354.1	Ortho Phosphorus					
365.1, 365.3	Ortho Phosphorus	Water	500 mL Plastic	≤6 °C; H_2SO_4; pH < 2	48 Hours	NA
9210, 9211	Nitrate	Water	1 L Plastic	≤6 °C; 1M Boric Acid	48 Hours	NA
		Solid/Other	250 mL Glass Jar	≤6 °C	48 Hours	NA
314.0, 9058	Perchlorate by IC	Water	250 mL Plastic or Glass	≤6 °C	28 Days	NA
6850, 331.0	Perchlorate by HPLC/MS/MS	Water	250 mL Plastic or Glass	≤6 °C	28 Days	60 days
6860, 330.0	Perchlorate by IC/ESI/MS/MS					
		Solid	4 oz. Wide-mouth jar	≤6 °C	28 Days	60 days
410.3, 410.4	Chemical Oxygen Demand (COD)	Water	250 mL Glass	≤6 °C; H_2SO_4; pH < 2	28 Days	NA
1664	Total Recoverable Oil and Grease	Water	1 L Glass	≤6 °C; H_2SO_4 or HCl; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	≤6 °C	28 Days	NA
9070A, 9071B	Total Recoverable Oil and Grease	Water	1 L Glass	≤6 °C; HCl; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	≤6 °C	28 Days	NA
ASTM D-854	Specific Gravity	Water	500 mL Plastic or Glass	None	None	

Note: 40 mL vials require caps with Teflon lined septum. All other containers require Teflon lined screw cap lids to minimize container contamination and loss of analyte.
*If samples are shipped to the laboratory in EnCore™ samplers, samples must be extruded and placed in sample containers within 48 hours of sample collection.

Sample Preservation Techniques and Holding Times

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times Extract
9030B, 9031	Sulfide	Water	1 L Glass	≤6 °C; NaOH; Zinc acetate; pH > 9	7 Days	NA
4500S[2]-D, E, F, or G		Solid/Other	125 mL Glass Jar	≤6 °C	7 Days	NA
2540 B, C, D	TDS, TSS, TS	Water	1 L Plastic	≤6 °C	7 Days	NA
160.4	Volatile solids (volatile residue)	Water	Plastic or glass	≤6 °C	7 Day	NA
9020B	TOX	Water	1 L Amber Glass	≤6 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	≤6 °C	28 Days	NA
9060A	TOC	Water	Glass	≤6°C; H_2SO_4 or HCL; pH < 2 if analyzed >2 hours after collection	2 hours, unless acidified	N/A
418.1	Total Petroleum Hydrocarbon (TPH)	Water	1 L Amber Glass	≤6 °C; HCl; pH < 2	28 Days	NA
1664A	TPH	Water	1 L Amber Glass	≤6°C; H_2SO_4 or HCl; pH < 2	28 Days	NA
8440	TPH	Solid/Other	125 mL Glass Jar	≤6 °C	28 Days	NA
9065, 9066	Total Recoverable Phenols	Water	1 L Glass	≤6 °C; H_2SO_4; pH < 4	28 Days	NA
420.1, 410.4		Solid	125 mL Glass Jar	≤6 °C	28 Days	NA
9040C 4500H[+]-B	pH	Water	125 mL Plastic	≤6 °C	ASAP	NA
2120B, C or E 180.1	Color, Turbidity	Water	500 mL Plastic	≤6 °C	48 Hours	NA
120.1, 9050A	Specific Conductance	Water	125 mL Plastic	≤6 °C	ASAP	NA

Note: 40 mL vials require caps with Teflon lined septum. All other containers require Teflon lined screw cap lids to minimize container contamination and loss of analyte.
*If samples are shipped to the laboratory in EnCore™ samplers, samples must be extruded and placed in sample containers within 48 hours of sample collection.

Sample Preservation Techniques and Holding Times

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times Extract
All radiochemical parameters except Rn-222 and tritium		Water	1 L Plastic (2 x 2 L Preferred)	HNO_3 ; pH < 2	180 Days	NA
		Solid/Other	250 mL Glass Jar		180 Days	NA
913.0	Radon 222	Water	125 mL Glass	None	72 Hours	NA
906.0	Tritium	Water	1 L Glass		180 Days	NA
		Solid/Other	Sample size will vary with moisture content		180 Days	NA
8015C/D	Petroleum Hydrocarbons (Diesel Range Organics)	Water[3]	2 x 1 L Amber Glass Bottle	≤6 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	≤6 °C	14 Days	40 Days
	Petroleum Hydrocarbons (Gasoline Range Organics)	Water[4]	3 x 40 mL Glass Vial	≤6 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	≤6 °C	14 Days	NA
5035A/ 8015C/D	Petroleum Hydrocarbons (Gasoline Range Organics)	Soil	4 x 40 mL Glass Vial	≤6 °C, 2 Vials $NaHSO_4$ 1 Vial CH_3OH, 1 Vial No Preservative	*14 days	NA
8021B	Halogenated Volatile Organics	Water[4]	3 x 40 mL Glass Vial	≤6 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	≤6°C	14 Days	NA
5035A/8021B	Halogenated Volatile Organics	Soil	4 x 40 mL Glass Vial	≤6°C, 2 Vials $NaHSO_4$ 1 Vial CH_3OH, 1 Vial No Preservative	*14 days	NA
8081B	Organochlorine Pesticides	Water[3]	4 L Amber Glass Bottle	≤6 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	≤6 °C	14 Days	40 Days
8082A	PCBs	Water[3]	4 L Amber Glass Bottle	≤6 °C	1 Year	1 Year
		Soil/Other	250 Glass Jar	≤6 °C	1 Year	1 Year

Note: 40 mL vials require caps with Teflon lined septum. All other containers require Teflon lined screw cap lids to minimize container contamination and loss of analyte.
*If samples are shipped to the laboratory in EnCore™ samplers, samples must be extruded and placed in sample containers within 48 hours of sample collection.

Sample Preservation Techniques and Holding Times

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times Extract
8141B	Organophosphorous Compounds	Water[3]	4 L Amber Glass Bottle	≤6 °C; NaOH or H_2SO_4; pH 5-8	7 Days	40 Days
		Soil/Other	250 Glass Jar	≤6 °C	14 Days	40 Days
8151A	Chlorinated Herbicides	Water[3]	4 L Amber Glass Bottle	≤6 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	≤6 °C	14 Days	40 Days
8260B/C	Volatile Organics by GC-MS	Water[1,2,4]	3 x 40 mL Glass Vial	≤6 °C; HCl; pH < 2	14 Days	NA
				≤6 °C; not acidified	7 Days	NA
		Soil/Other	125 mL Glass Jar	≤6 °C	14 Days	NA
5035A/ 8260B/C	Volatile Organics by GC-MS	Soil	4 x 40 mL Glass Vial	≤6 °C, 2 Vials $NaHSO_4$ 1 Vial CH_3OH, 1 Vial No Preservative	*14 days	NA
8270C/D	Semivolatile Organics by GC-MS	Water[3]	4 L Amber Glass Bottle	≤6 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	≤6°C	14 Days	40 Days
8280B	Polychlorinated Dioxins and Furans by HRGC/LRMS	Water[3]	4 L Amber Glass Bottle	≤6 °C	30 Days	45 Days
		Soil/Other	250 mL Glass Jar	≤6 °C	30 Days	45 Days
8290A	Dioxins and Furans By HRGC/HRMS	Water[3]	4 L Amber Glass Bottle	≤6 °C	30 Days	45 Days
		Soil/Other	250 mL Glass Jar	≤6 °C	30 Days	45 Days
1613	Dioxins and Furans by Isotope Dilution HRGC/HRMS	Water[3]	Amber Glass	≤6 °C	1 Year	1 Year
		Solid	Amber Glass Jar	≤6 °C	1 Year	1 Year
1668A	PCB Congeners by HRGC/HRMS	Water[3]	Amber Glass	≤6 °C,	1 Year	1 Year
		Solid	Amber Glass Jar	≤6 °C	1 Year	1 Year

Note: 40 mL vials require caps with Teflon lined septum. All other containers require Teflon lined screw cap lids to minimize container contamination and loss of analyte.
*If samples are shipped to the laboratory in EnCore™ samplers, samples must be extruded and placed in sample containers within 48 hours of sample collection.

Sample Preservation Techniques and Holding Times

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times Extract
1694	PPCP	Water[3]	Amber Glass	≤6 °C	7 Days	40 Days
		Solid	Amber Glass Jar	≤6 °C	7 Days	40 Days
8318A	N-Methylcarbamate Pesticides by HPLC	Water	4 L Amber Glass Bottle	≤6 °C; 0.1 N $ClCH_2CO_2H$, pH 4 - 5	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	≤6 °C	7 Days	40 Days
8330B	Nitroaromatics and Nitramines by HPLC	Water	4 L Amber Glass Bottle	≤6 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	≤6 °C	14 Days	40 Days
610, 8310	PAHs by HPLC	Water[3]	Amber Glass/Teflon lined cap	≤6 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	<6 °C	14 Days	40 Days
TO-13A	PAHs in Filter Cartridges	PUF, Tenax, or XAD-2 Filter Cartridge		≤6 °C	7 Days	40 Days
TO-14A, TO-15		VOC in Air	SUMMA® Canister		30 Days	
8321A (modified)	High Explosives by LC/MS/MS	Water	Amber Glass/Teflon lined cap	≤6 °C	7 Days	40 Days
		Solid	Amber Glass/Teflon lined cap	≤6 °C	14 Days	40 Days

[1] If vinyl chloride, styrene, or 2-chloroethylvinylether are analytes of interest, collect a second set of samples without acid preservative and analyze within 7 days.

[2] If acrolein and acrylonitrile are analytes of interest, adjust to pH 4-5.

[3] If residual chlorine is present, preserve with 80 mg of sodium thiosulfate per liter of water.

[4] If residual chlorine is present, preserve with 10 mg of sodium thiosulfate per 125 mL of water.

Note: 40 mL vials require caps with Teflon lined septum. All other containers require Teflon lined screw cap lids to minimize container contamination and loss of analyte.
*If samples are shipped to the laboratory in EnCore™ samplers, samples must be extruded and placed in sample containers within 48 hours of sample collection.

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Appendix C

Data Reporting Requirements

If any quality control (QC) samples are analyzed using a different initial calibration (ICAL) than that of the field samples, the laboratory must include a calibration report for the calibration affecting the QC samples. This calibration data shall only be used to evaluate the QC samples, and only if the QC samples fail to meet recovery or relative percent difference (RPD) acceptance criteria. The laboratory is not required to report calibration data associated with QC samples from another sample delivery group.

If required data is not present contact the laboratory to request an amended report. Documentation may include the following, as appropriate.

Gas Chromatography/Mass Spectrometry (GC/MS)

- Case narrative
- Instrument tuning data
- ICAL data
- Applicable calibration verification data
- Continuing calibration check data
- Instrument and preparation blank data
- Surrogate data
- Internal standard (IS) performance data
- Matrix spike/matrix spike duplicate (MS/MSD) data
- Laboratory control sample (LCS) data
- Sample results and analytical data for the requested target analytes, including results from dilutions, if analyzed
- Identification and data for any sample tentatively identified compounds
- Instrument run logs
- Analysis Request and Chain of Custody (ARCOC) and shipping documents
- Login worksheet
- Laboratory replicate data, if analyzed

Dioxins and Furans by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)

- Case narrative
- Column performance check data
- ICAL data
- Applicable calibration verification data
- Continuing calibration check data
- Preparation blank data
- Labeled compound data
- Ongoing precision and recovery (OPR) data
- Ion abundance ratio data
- Sample results and analytical data for the requested target analytes, including results from dilutions, if analyzed
- Instrument run logs
- ARCOC and shipping documents
- Login worksheet
- Laboratory replicate data, if analyzed

Gas Chromatography (GC) and High-Performance Liquid Chromatography (HPLC)

- Case narrative
- ICAL calibration data, including secondary column, if appropriate
- Applicable calibration verification data
- Continuing calibration check data
- Instrument and preparation blank data
- Surrogate data
- MS/MSD data
- LCS data
- Sample results and analytical data for the target analytes, including data from dilutions, if analyzed
- Confirmation data and RPD between the results
- Instrument run logs
- ARCOC and shipping documents
- Login worksheet
- Laboratory replicate data, if analyzed

Polychlorinated Biphenyl (PCB) Congeners, EPA Method 1668A

- Case Narrative
- ICAL data, including relative retention time (RRT) windows
- Calibration verification data including ion abundance ratios and RRTs
- Preparation blank data
- OPR data
- Clean-up standard data
- Labeled compound data
- Sample results and analytical data for the requested target analytes, including results from dilutions, if analyzed
- Ion abundance ratio for all detected sample results, labeled compounds, and clean-up standards
- RRTs for all detected sample results, labeled compounds, and clean-up standards
- Instrument run logs
- Sample preparation data
- Lipid data (tissue samples only)
- ARCOC and shipping documents
- Login worksheet

High Explosives (HE) and Perchlorate by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)

- Case narrative
- ICAL data
- Applicable calibration verification data
- Initial calibration blank (ICB) data
- Continuing calibration check data

- Continuing calibration blank (CCB) data
- Low level calibration verification (CRI) [1] data
- Instrument and preparation blank data
- Surrogate data (HE only)
- IS or Method of Standard Addition performance data
- MS/MSD data
- LCS data
- Retention time data
- Isotope Ratio data (perchlorate only)
- Sample results and analytical data for the requested target analytes, including results from dilutions, if analyzed
- Instrument run logs
- ARCOC and shipping documents
- Login worksheet
- Laboratory replicate data, if analyzed

Inorganic

- Case narrative
- ICAL data
- Applicable calibration verification data
- ICB data
- Continuing calibration data
- CCB data
- Instrument tuning data
- Instrument and preparation blank data
- MS data
- LCS data
- Laboratory replicate data
- Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and inductively coupled plasma-mass spectrometry (ICP-MS) interference check sample data
- ICP SD data
- PQL verification (CRA/CRI/CRDL)[2]
- Sample results and analytical data, including data from dilutions, if analyzed
- Instrument run logs
- ARCOC and shipping documents
- Login worksheet

[1] CRI = reporting limit verification (RLV) for LC/MS/MS, ICP-AES and ICP-MS methods.
[2] CRA = RLV for atomic absorption (AA) methods.
CRI = RLV for LC/MS/MS, ICP-AES, and ICP-MS methods.
CRDL = RLV for cyanide methods.

Radiochemistry

- Case narrative
- Instrument and preparation blank data
- Applicable calibration verification data
- MS data
- LCS data
- Laboratory replicate data
- Sample results
- Carrier or chemical tracer data
- Instrument run logs
- ARCOC and shipping documents
- Login worksheet
- Control Charts

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Appendix D

Surrogate Recovery Limits

Guidelines for Surrogate Recovery Limits

Volatile Organics – Water	**Mean**	**Lower Limit**	**Upper Limit**
1,2-Dichloroethane-d4	95	72	119
4-bromofluorobenzene	98	76	119
Dibromofluoromethane	100	85	115
Toluene-d8	102	83	120
Volatile Organics – Solid			
4-bromofluorobenzene	101	84	118
Toluene-d8	100	84	116
Semivolatile Organics – Water			
2-fluorobiphenyl	79	48	112
Terphenyl-d14	92	51	135
2,4,6-Tribromophenol	82	42	124
2-Fluorophenol	63	19	108
Nitrobenzene-d5	76	41	111
Semivolatile Organics – Solid			
2-fluorobiphenyl	72	43	103
Terphenyl-d14	78	32	125
2,4,6-Tribromophenol	80	36	126
2-Fluorophenol	70	37	104
Phenol-d5/d6	71	40	102
Nitrobenzene-d5	69	37	102
Pesticides – Water			
Decachlorobiphenyl	83	32	135
Tetrachlorometaxylene (TCMX)	81	25	138
Pesticides – Solid			
Decachlorobiphenyl	94	56	132
TCMX	97	69	124
Polychlorinated Biphenyl (PCB) – Water			
Decachlorobiphenyl	88	42	133
PCB – Solid			
Decachlorobiphenyl	91	58	125
High Explosives (HE) – Water			
3,4-Dinitrotoluene	86	33	139
2-Methyl-4-nitroaniline	86	33	139
1,4-Dintrobenzene	86	33	139
1,2-Dintrobenzene	86	33	139
HE – Solid			
3,4-Dinitrotoluene	98	56	140
2-Methyl-4-nitroaniline	98	56	140
1,4-Dintrobenzene	98	56	140
1,2-Dintrobenzene	98	56	140

Appendix E

Gas Chromatography/Mass Spectrometry (GC/MS) Internal Standards

Laboratories may vary the compounds calculated off of any internal standard (IS) and should identify within the report which compounds were calculated from each IS. If this information is not readily available the following tables may be used as guidelines.

GC/MS Volatile Organic Analysis Internal Standard Tables

Fluorobenzene

Chloromethane	Vinyl Chloride	Bromomethane	Chloroethane
Acetone	1,1-Dichloroethene	Methylene Chloride	Carbon Disulfide
1,1-Dichloroethane	Trans-1,2-Dichloroethene	2-Butanone	2,2-Dichloropropane
Cis-1,2-Dichloroethene	Chloroform	Bromochloromethane	1,1,1-Trichloroethane
1,1-Dichloropropene	Carbon Tetrachloride	1,2-Dichloroethane	Benzene
Trichloroethene	1,2-Dichloropropane	Bromodichloromethane	Dibromomethane
4-Methyl-2-pentanone	Cis-1,3-Dichloropropene	Trichlorofluoromethane	Acetonitrile
Acrolein	Acrylonitrile	n-Butyl alcohol	2-Chloro-1,3-butadiene
Dichlorodifluoromethane	1,4-Dioxane	Ethyl acetate	Iodomethane
Isobutyl alcohol	Methacrylonitrile	Methyl methacryalate	Methyl isobutyl ketone
Propionitrile	Trichlorofluoromethane		

Chlorobenzene-d5

Toluene	Trans-1,3-Dichloropropene	1,1,2-Trichloroethane	2-Hexanone
1,3-Dichloropropane	Tetrachloroethene	Chlorodibromomethane	1,2-Dibromoethane
Chlorobenzene	1,1,1,2-tetrachloroethane	Ethylbenzene	m,p-Xylenes
o-Xylene	Styrene	Bromoform	1,1,2,2-Tetrachloroethane
Ethyl methacrylate			

1,4-Dichlorobenzene-d4

1,2,3-Trichloropropane	Bromobenzene	1,2-Dibromo-3-chloropropane	1,2-Dichlorobenzene
1,3-Dichlorobenzene	1,4-Dichlorobenzene	Hexachlorobutadiene	Napthalene
Pentachloroethane	1,2,4-Trichlorobenzene		

GC/MS Semivolatile Organic Analysis Internal Standard Tables

1,4-Dichlorobenzene-d4

2-Fluorophenol	Phenol-d5	2-Chlorophenol-d4	1,2-Dichlorobenzene-d4
Phenol	Bis (2-Chloroethyl) ether	2-Chlorophenol	1,3-Dichlorobenzene
1,4-Dichlorobenzene	1,2-Dichlorobenzene	2-Methylphenol	2,2'-oxybis(2-Chloropropane
4-Methylphenol	N-Nitroso-di-n-propylamine	Hexachloroethane	Pyridine
Acetophenone	Aniline	Methyl metanesulfonate	N-Nitrosodiethylamine
N-Nitrosodimethylamine	N-Nitrosomethylethylamine	N-Nitrosomorpholine	N-Nitrosopiperidine
N-Nitrosopyrrolidine			

Naphthalene-d8

Nitrobenzene-d5	Nitrobenzene	Isophorone	2-Nitrophenol
2,4-Dimethylphenol	Bis (2-Chloroethoxy) methane	2,4-Dichlorophenol	1,2,4-Trichlorobenzene
Naphthalene	4-Chloroaniline	Hexachlorobutadiene	4-Chloro-3-methylphenol
2-Methylnaphthalene	Benzoic Acid	2.6-Dichlorophenol	Hexachloropropene
N-Nitrosodi-n-butylamine	Safrole		

Acenaphthene-d10

2,4,6-Tribromophenol	2-Fluorobiphenyl	Hexachlorocyclopentadiene	2,4,6-Trichlorophenol
2,4,5-Trichlorophenol	2-Chloronaphthalene	2-Nitroaniline	Dimethylphthalate
Acenaphthylene	2,6-Dinitrotoluene	3-Nitroaniline	Acenaphthene
2,4-Dinitrophenol	4-Nitrophenol	Dibenzofuran	2,4-Dinitrotoluene
Diethylphthalate	4-Chlorophenyl phenyl ether	Fluorene	4-Nitroaniline
2-sec-Butyl-2,6-dinitrophenol	Isosafrole	N-Nitro-o-toluidine	Pentachlorophenol
1,2,4,5-Tetrachlorobenzene	2,3,4,6-Tetrachlorophenol		

Phenanthrene-d10

2,4-Dinitro-2-methylphenol	N-Nitrosodiphenylamine	4-Bromophenyl phenyl ether	Hexachlorobenzene
Pentachlorophenol	Phenanthrene	Anthracene	Carbazole
Di-n-butylphthalate	Fluoranthene	Methapyrilene	Pentachloronitrobenzene
Phenacetin	Pronamide		

Chrysene-d12

Pyrene	Butylbenzylphthalate	3,3'-Dichlorobenzidine	Benzo(a)anthracene
Chrysene	Bis(2-Ethylhexl)phthalate	2-Acethylaminofluorene	Chlorobenzilate

Perlene-d12

Di-n-octylphthalate	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene	Dibenz(a,h)anthracene	Benzo(g,h,i)perylene	Hexachlorophene
3-Methylcholanthrene			

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Appendix F

Laboratory Control Limits

Organic Laboratory Control Sample (LCS) Criteria Guidelines (Volatile Compounds)

Volatile Compound	CAS #*	Water			Solid		
		Ave	Low	High	Ave	Low	High
Acetone	67-64-1	91	39	142	88	19	158
Benzene	71-43-2	102	81	122	99	73	126
Bromobenzene	108-86-1	100	76	124	93	66	121
Bromochloromethane	74-97-5	97	65	129	99	71	127
Bromodichloromethane	75-27-4	98	76	121	100	72	128
Bromoform	75-25-2	99	69	128	96	56	137
Bromomethane	74-83-9	88	30	146	95	31	159
2-Butanone	78-93-3	91	32	150	94	29	159
n-Butylbenzene	104-51-8	103	69	137	101	65	138
sec-Butylbenzene	135-98-8	100	72	127	97	63	132
tert-Butylbenzene	98-06-6	99	70	129	99	65	132
Carbon disulfide	75-15-0	100	37	162	103	47	159
Carbon tetrachloride	56-23-5	102	66	138	100	67	133
Chlorobenzene	108-90-7	102	81	122	99	75	123
Chloroethane	75-00-3	99	62	135	98	39	157
Chloroform	67-66-3	100	63	136	98	72	124
Chloromethane	74-87-3	83	39	127	90	51	129
2-Chlorotoluene	95-49-8	100	73	126	98	69	128
4-Chlorotoluene	106-43-4	101	74	128	100	73	126
Dibromochloromethane	124-48-1	96	58	133	98	66	130
1,2-Dibromo-3-chloropropane	96-12-8	91	50	132	87	40	135
1,2-Dibromoethane	106-93-4	100	80	121	97	70	124
Dibromomethane	74-95-3	101	76	125	100	73	128
1,2-Dichlorobenzene	95-50-1	96	71	122	97	74	119
1,3-Dichlorobenzene	541-73-1	100	75	124	98	72	124
1,4-Dichlorobenzene	106-46-7	99	74	123	98	72	125
Dichlorodifluoromethane	75-71-8	93	31	155	85	34	136
1,1-Dichloroethane	75-34-3	101	69	133	99	73	125
1,2-Dichloroethane	107-06-2	100	69	132	104	72	137
1,1-Dichloroethene	75-35-4	99	68	130	100	65	136
cis-1,2-Dichloroethene	156-59-2	99	72	126	96	67	125
trans -1,2-Dichloroethene	156-60-5	99	60	139	100	66	134
1,2-Dichloropropane	78-87-5	100	75	125	95	71	119
1,3-Dichloropropane	142-28-9	100	73	126	100	76	123
2,2-Dichloropropane	594-20-7	103	69	137	101	67	134
1,1-Dichloropropene	563-58-6	102	73	132	102	70	135
cis-1,3-Dichloropropene	10061-01-5	100	69	131	99	72	126
trans-1,3-Dichloropropene	10061-02-6	98	53	142	96	65	127
Ethylbenzene	100-41-4	100	73	127	101	74	127
Hexachlorobutadiene	87-68-3	97	51	142	98	53	142

Organic LCS Criteria Guidelines (Volatile Compounds) (concluded)

Volatile Compound	CAS #*	Water			Solid		
		Ave	Low	High	Ave	Low	High
2-Hexanone	591-78-6	92	56	128	97	47	146
Isopropylbenzene	98-82-8	101	75	127	103	77	129
4-Isopropyltoluene	99-87-6	102	73	131	104	75	133
Methylene chloride	75-09-2	96	53	140	97	54	141
4-Methyl-2-pentanone	108-10-1	96	58	134	97	47	147
Naphthalene	91-20-3	96	54	138	84	40	127
n-Propylbenzene	103-65-1	101	72	129	99	63	135
Styrene	100-42-5	100	65	134	101	74	128
1,1,1,2-Tetrachloroethane	630-20-6	105	81	129	100	74	125
1,1,2,2-Tetrachloroethane	79-34-5	96	63	128	93	54	131
Tetrachloroethene	127-18-4	96	44	149	103	67	139
Toluene	108-88-3	100	77	122	99	71	127
1,2,3-Trichlorobenzene	87-61-6	99	57	142	97	62	133
1,2,4-Trichlorobenzene	120-82-1	100	66	134	98	65	131
1,1,1-Trichloroethane	71-55-6	100	67	132	101	68	133
1,1,2-Trichloroethane	79-00-5	100	75	125	95	62	127
Trichloroethene	79-01-6	96	44	149	103	67	139
1,2,3-Trichloropropane	96-18-4	98	73	124	97	63	130
1,2,4-Trimethylbenzene	95-63-6	103	74	132	98	65	131
1,3,5-Trimethylbenzene	108-67-8	102	74	131	99	65	133
Vinyl chloride	75-01-4	99	50	147	92	58	126
o-Xylene	95-47-6	100	80	121	101	77	125

*chemical abstract service number

Organic LCS Criteria Guidelines (Semivolatile Compounds)

Semivolatile Compound	CAS #*	Water			Solid		
		Ave	Low	High	Ave	Low	High
Polynuclear Aromatics							
2-Methynaphthalene	91-57-6	75	46	104	77	47	107
Acenaphthene	83-32-9	78	47	108	77	46	108
Acenaphthylene	208-96-8	79	50	107	76	44	107
Anthracene	120-12-7	83	54	112	80	53	107
Benz(a)anthracene	56-55-3	83	56	109	82	52	111
Benzo(b)fluoranthene	205-99-2	82	45	118	80	45	114
Benzo(k)fluoranthene	207-08-9	85	45	124	84	45	123
Benzo(g,h,i)perylene	191- 24-2	81	38	123	82	38	126
Benzo(a)pyrene	50-32-8	81	53	110	81	50	111
Chrysene	218-01-9	82	55	109	83	53	112
Dibenz(a,h)anthracene	53-70-3	85	42	127	83	41	125
Fluoranthene	206-44-0	85	54	116	84	54	114
Fluorene	86-73-7	81	50	112	78	49	108
Indeno(1,2,3-cd)pyrene	193-39-5	84	43	125	80	38	121
Naphthalene	91-20-3	71	39	102	73	40	107
Phenanthrene	85-01-8	84	51	117	80	50	110
Pyrene	129-00-0	89	49	128	84	46	123
Phenolic/Acidic							
2,4-Dichlorophenol	120-83-2	76	48	105	77	45	110
2, 4-Dimethylphenol	105-67-9	69	28	109	67	32	103
2,4-Dinitrophenol	51-28-5	76	14	138	73	13	132
2-Chlorophenol	95-57-8	71	37	106	75	44	106
2-Methylphenol	95-48-7	73	38	109	72	40	104
3-Methylphenol	108-39-4	71	32	110	74	41	107
4-Methylphenol	106-44-5	71	32	110	74	41	107
2-Nitrophenol	88-75-5	76	39	113	76	42	111
4,6-Dinitro-2-methylphenol	534-52-1	85	40	130	83	29	137
4-Chloro-3-methylphenol	59-50-7	79	47	111	80	46	113
Pentachlorophenol	87-86-5	78	38	117	72	25	119
4-Nitrophenol	100-02-7				77	17	138
Phenol	108-95-2				70	39	100
Basic							
3,3'-Dichlorobenzidine	91-94-1	65	19	111			
4-Chloroaniline	106-47-8	62	15	109			
Phthalate Esters							
Bis(2-ethylhexyl) phthalate	117-81-7	84	42	126	87	47	127
Butyl benzyl phthalate	85-68-7	81	46	116	86	49	123
Di-n-butyl phthalate	84-74-2	85	54	116	83	56	110
Di-n-octyl phthalate	117-84-0	87	37	137	86	41	132

Organic LCS Criteria Guidelines (Semivolatile Compounds) (concluded)

Semivolatile Compound	CAS #*	Water			Solid		
		Ave	Low	High	Ave	Low	High
Polynuclear Aromatics							
Diethyl phthalate	84-66-2	79	41	118	82	50	114
Dimethyl phthalate	131-11-3	76	25	127	80	49	110
Nitrosoamines							
N-Nitrosodimethylamine	62-75-9	68	26	110	66	18	114
N-Nitrosodiphenylamine	86-30-6	80	48	111	82	49	116
N-Nitroso-di-n-propylamine	621-64-7	81	34	128	77	40	114
Chlorinated Aliphatics							
Bis(2-chloroethoxy) methane	111-91-1	76	46	107	76	43	108
Bis(2-chloroethyl) ether	111-44-4	73	37	110	71	38	105
Bis(2-chloroisopropyl) ether	108-60-1	78	26	131	68	21	115
Hexachlorobutadiene	87-68-3	65	27	103	78	40	117
Hexachloroethane	67-72-1	61	28	94	72	37	110
Halogenated Aromatics							
1,2,4-Trichlorobenzene	120-82-1	72	37	107	77	44	111
1,2-Dichlorobenzene	95-50-1	67	33	102	71	45	97
1,3-Dichlorobenzene	541-73-1	65	32	98	70	39	100
1,4-Dichlorobenzene	106-46-7	65	32	98	69	35	103
2-Chloronaphthalene	91-58-7	77	49	104	75	45	105
4-Bromophenyl phenyl ether	101-55-3	83	52	113	82	46	117
4-Chlorophenyl phenyl ether	7005-72-3	81	50	111	80	47	112
Hexachlorobenzene	118-74-1	82	52	112	83	47	118
Nitroaromatics							
2,4-Dinitrotoluene	121-14-2	84	51	118	82	48	116
2,6-Dinitrotoluene	606-20-2	83	49	117	80	48	112
2-Nitroaniline	88-74-4	82	48	115	81	44	118
3-Nitroaniline	99-09-2	73	19	126	69	27	110
4-Nitroaniline	100-01-6	77	36	118	74	34	113
Nitrobenzene	98-95-3	77	44	109	77	41	113
Neutral Aromatics							
Carbazole	86-74-8	83	48	117	80	44	117
Dibenzofuran	132-64-9	80	54	107	77	51	103
Others							
Benzyl alcohol	100-51-6	71	30	112	71	19	123
Isophorone	78-59-1	81	50	112	77	43	111

*chemical abstract service number

Organic LCS Criteria Guidelines
(Pesticides and Polychlorinated Biphenyl [PCB])

Pesticide	CAS #*	Water			Solid		
		Ave	Low	High	Ave	Low	High
Aldrin	309-00-2	83	27	138	93	47	140
α-benzene hexachloride (BHC)	319-84-6	94	60	128	93	62	125
β-BHC	319-85-7	96	66	126	95	62	127
δ-BHC	319-86-8	91	46	136	94	57	130
γ-BHC (Lindane)	58-89-9	82	27	137	91	59	123
α-Chlordane	5103-71-9	93	63	123	92	63	121
4,4'- Dichlorodiphenyldichloroethane (DDD)	72-54-8	88	27	149	81	28	135
4,4'- Dichlorodiphenyldichloroethylene (DDE)	72-55-9	87	33	140	97	68	126
4,4'- Dichlorodiphenyltrichloroethane (DDT)	50-29-3	92	47	138	92	45	140
Dieldrin	60-57-1	95	62	129	96	67	125
Endosulfan I	959-98-8	80	49	111	74	14	133
Endosulfan II	33213-65-9	79	28	130	89	37	141
Endosulfan sulfate	1031-07-8	96	54	137	99	62	135
Endrin	72-20-8	95	56	134	97	61	133
Endrin aldehyde	7421-93-4	96	56	137	92	37	147
Endrin ketone	53494-70-5	102	77	127	100	66	134
Heptachlor	76-44-8	87	42	131	96	51	140
Heptachlor epoxide	1024-57-3	96	62	131	98	66	130
4,4’-Methoxychlor	72-43-5	103	56	150	100	57	143
PCB							
Aroclor – 1016	12674-11-2	85	25	144	90	41	138
Aroclor – 1260	11096-82-5	87	30	146	96	61	131

*chemical abstract service number

Organic LCS Criteria Guidelines (Nitroaromatics and Nitramines)

Nitroaromatics and Nitramines	CAS #*	Water			Solid		
		Ave	Low	High	Ave	Low	High
2-Amino-4,6-Dinitrotoluene (2-Am-DNT)	355-72-78-2	87	59	115	102	80	124
4-Amino-2,6-Dinitrotoluene (4-Am-DNT)	1946-51-0	96	56	137	101	79	124
1,3-Dinitrobenzene (DNB)	99-65-0				101	79	124
2,4-Dinitrotoluene (24DNT)	121-14-2	83	12	154	98	36	161
2,6-Dinitrotoluene (26DNT)	606-20-2				100	77	122
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	88	40	136	103	72	134
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8	85	17	153			
Nitrobenzene (NB)	98-95-3				96	39	154
2-Nitrotoluene (2NT)	88-72-2				97	39	156
3-Nitrotoluene (3NT)	99-08-1	80	15	146	95	32	159
4-Nitrotoluene (4NT)	99-99-0	80	16	144	101	77	124
Pentaerythritol tetranitrate (PETN)							
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	89	47	131	100	74	126
Trinitrobenzene (135TNB)	99-35-4				95	34	156
2,4,6-Trinitrotoluene (TNT)	118-96-7				95	17	173

*chemical abstract service number

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Appendix G

Mass Spectra Acceptability

Mass Spectra Acceptability

Ideal spectral identification of a target analyte by a mass spectrometer data system is performed by comparing three characteristic ions (i.e., a primary or quantitation ion, a secondary ion, and a tertiary ion) from one mass spectrum to the same characteristic ions in the reference mass spectrum. The three characteristic ions from the mass spectrum are defined as the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. In most cases, ions with the greatest abundance are used for evaluation; however, if target analytes suffer from coelution, interferences may dictate the use of less abundant ions for evaluation. Some analytes generate a mass spectrum that is of such a simple nature that a tertiary ion is not sufficiently abundant (e.g., low molecular weight analytes and analytes that do not fragment sufficiently upon electron impact ionization). In this case, two ions are used for identification.

For evaluation of analyte spectra, all of the following factors should be considered before an acceptability judgment is made.

Retention Times (RT)

The intensities of the primary, secondary, and, if applicable, tertiary ions at the established RT of the target analyte are shown in the extracted ion current profiles (EICP). The RTs for the secondary and tertiary ion profiles should be the same as the primary ion. Depending on peak shape and chromatographic interferences, the RTs could differ by a few hundredths of a minute; however, the RTs between primary and secondary ions should not vary by more than 0.03 minutes.

Relative Retention Times (RRT)

The RRT of the target analyte in the sample should agree to within 0.06 RRT units of the same analyte in the reference standard (either the midpoint standard of the initial calibration or the daily continuing calibration verification).

Ion Ratios

The most intense ion in a spectrum is assigned a relative abundance of 100 and is known as the base peak. The intensities of all other ions in the spectrum are compared to the intensity of the base peak to obtain an intensity ratio (or ion ratio). The ion ratios for the three characteristic ions from the sample spectrum are compared to the ion ratios for the same ions from the reference spectrum. Relative intensities should agree to within ±30%. For example, an ion that has an abundance of 50% when compared to the base peak in the reference spectrum will have a range of 20-80% as its acceptance criteria for that same ion in the sample spectrum.

Ion ratio evaluation is performed by the laboratory. Gas chromatography/mass spectrometry data systems automatically flag target analytes with a “Q” or a “#” on the quantitation report when ratio comparison criteria are exceeded. Because interferences and varying instrument conditions can affect relative abundances, the presence of a Q flag does not necessarily indicate an invalid identification; however, a Q flag in addition to other guideline failures may result in the need for additional data in order to make an acceptability judgment.

Visual Comparison of Mass Spectra

The sample spectrum should be visually compared to the reference spectrum for pattern similarity. When a peak elutes in a discrete manner and the reference spectrum was obtained under similar conditions as the sample spectrum, the mass spectral pattern from the sample will be similar to the mass spectral pattern of the reference spectrum. If coelution occurs or a high level of background is present, the partial or total spectral pattern from the reference spectrum should be visible within the spectral pattern of the sample spectrum, with sample ion ratios emulating the reference ion ratios, depending on the inherent complexity of the analyte spectrum.

Identification of Target Analytes Present in Samples at Low Levels

Spectra from target analytes that are detected at levels around the established method detection limit (MDL) should be examined carefully for the presence of secondary and, if applicable, tertiary ions. For a qualitative identification to be made, all ions used by the instrument method should be present. Because background noise, column bleed, and interferences can hamper identification, correct subtraction is important to identification. Supplemental data may be required from the laboratory in order to properly separate chromatographic interferences. These data may include a library search with listed spectral fit (or match) quality (i.e., a "Q report") or additional EICPs displaying interfering ions for RT comparison.

Interferences

Identification of target analytes is hampered when sample components are not resolved chromatographically (i.e., there is co-elution of non-target and/or target analytes) and produce mass spectra containing ions from more than one analyte. When gas chromatography (GC) peaks, EICPs, or spectra show evidence of interference (e.g., GC or EICP peak appears broadened with shoulders, obviously overlapping peaks are present, or extraneous ions are present in the spectrum), supplemental data may be required from the laboratory in order to properly evaluate analyte spectra. These data may include a Q report or additional EICPs displaying interfering ions for RT comparison.

Guidelines for Use of Supplemental Data

Q Report

If a target analyte is detected by the mass spectrometer data system and its identification is questionable, the mass spectrum for that analyte may be subjected to a computer comparison against a library of established mass spectra (i.e., a "library search"). This search generates a Q report that shows the mass spectrum being searched, the compounds in the library whose spectra most closely matches the mass spectrum being searched, and a Chemical Abstract Services (CAS) number and a match quality rating from 1-100 (with 100 being a perfect match fit) for each of those compounds. A Q report obtained for a spectrum at the RT of the analyte in question can sometimes help identify that analyte. Ideally, if a data system identifies a target analyte using the identification parameters discussed (i.e., RT, RRT, and major ion intensity ratios), a library search of the analyte should yield concurrent results with a match rating of >75. The following variables affect match quality ratings:

The Analyte Concentration

For identification of an analyte of low concentration (i.e., detections at or just above the MDL), ions of >50% relative intensity in library spectrum should be present in sample spectrum. If minor ions in the analyte spectrum are absent due to low concentration, the match quality rating may be low.

The Nature of the Spectrum

If the spectrum of the analyte in question is relatively complex (i.e., the spectrum contains multiple ions of >50% relative intensity), match quality ratings will generally be higher. If the spectrum of the peak in question only yields two or three ions within scanning range, separation from interferences and background is sometimes not possible, making match quality ratings lower. Also, if the spectrum of the analyte in question has one or more ions common to known contaminants, misidentification will be more common (e.g., acetone with its primary ion of 43 is sometimes hard to distinguish from early eluting, low molecular weight hydrocarbons that have the same ion as their primary ions).

The presence of one or more interferences can affect match quality ratings. One dominant interference can yield spectral match quality ratings that are high but whose best match is primarily due to the interfering non-target analyte, not the analyte in question. Multiple interferences will usually yield match quality ratings that are low due to the inability of the software to make a match without a dominant pattern.

The Conditions Under Which the Library Spectrum is Obtained

If spectral quality matches are low, especially for the target analyte in question, the library spectra for the analyte in question should be considered suspect. Mass spectra in established libraries are normally generated under wider scan ranges than are dictated in methods. Low molecular weight analytes in the library may have one or more characteristic ions below the scanning range of the environmental analytical method, rendering the spectra in the low molecular weight range only partially comparable. If it is obvious that the library mass spectrum for an analyte was obtained under different scanning conditions as the sample spectrum, match quality ratings may be reduced.

Additional EICPs

The review of EICPs of all ions of >50% relative abundance, including the primary, secondary, and tertiary ions of the analyte in question as well as ions from interfering analytes, is one way to determine interference separability and abundance contribution. If some or all of the ions from an interfering analyte (i.e., those not contained in the target analyte in question) maximize at the same RT as the target analyte in question, it is possible that interfering ions are contributing abundance to the analyte in question. Also, if peak shape is variable, it is possible that two or more compounds are co-eluting and are contributing a range of ions and overlapping chromatographic peak shapes. If EICPs show that the characteristic ions from the analyte in question elute at the same RT and that RT differs from the EICPs of the ions from interfering compounds, then it is possible that the target analyte in question is present.

SANDIA NATIONAL LABORATORIES
LONG TERM STEWARDSHIP DEPARTMENT (4142)

SOIL VAPOR MONITORING
FIELD OPERATING PROCEDURE

FOP 08-22
Revision 3

Approved: _____ **Date:** 4/29/2014
Robert Ziock, SME

Approved: _____ **Date:** 4/29/14
Don Schofield, Field Support Operations Project Lead

Approved: _____ **Date:** 5/16/14
Pamela Puissant, Manager

Author: *How frequently does this document need to be reviewed and/or revised?*	*Every three years*
Manager: *Does this document need to be tracked?*	*Yes*

EFFECTIVE DATE: _____

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Revision History

Revision	Effective Date	Summary of Changes
0	1/27/2009	Original Issue
1	5/27/09	Section 6.3 – "Quality Control Sample Equipment Setup and Sampling Procedure" added.
2	6/09/2011	The rewrite makes FOP not specific to the CAMU. It now applies to soil vapor sampling at any SNL/NM site. Site-specific information for CAMU, CWL, MWL, and TA-V included in the attachments.
3		Updates include methane gas monitoring and attachment for TA-III Classified Waste Landfill. On-the-Job Training, Authorized User List, and Tailgate Safety Briefing attachments removed. Table B-1 and purge time requirements removed from Attachment B. Updated Attachment C to reflect approval of the LTMMP.

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ACRONYMS

AR/COC	Analysis Request/Chain of Custody
AOP	administrative operation procedure
CAMU	Corrective Action Management Unit
CLWL	TA-III Classified Waste Landfill
CWL	Chemical Waste Landfill
ES&H	Environment Safety & Health
ft	foot/feet
FOP	field operating procedure
GFCI	ground fault circuit interrupter
Hg	mercury
In.	inch/inches
LEL	lower explosive limit
LTMMP	Long-Term Monitoring and Maintenance Plan
MWL	Mixed Waste Landfill
NMED	New Mexico Environment Department
OJT	on-the-job training
PCC	Post-Closure Care
PHS	primary hazard screening
PID	photoionization detector
psi	pounds per square inch
QA/QC	quality assurance / quality control
SAP	sampling and analysis plan
SIH	standard industrial hazards
SMO	Sample Management Office
SNL/NM	Sandia National Laboratories/New Mexico
TA	Technical Area
TEDS	Training and Employee Development System
THA	task hazard analysis
VOC	volatile organic compound

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1.0 PURPOSE, SCOPE, AND OWNERSHIP

Purpose

The purpose of this field operating procedure (FOP) is to provide guidelines and procedures for soil vapor monitoring at different Sandia National Laboratories/New Mexico (SNL/NM) sites. Soil vapor monitoring can consist of taking *in situ* real-time measurements and/or collecting samples. Different sites are subject to different regulatory requirements and this is reflected in the type of monitoring that is performed. This procedure shall be used, as applicable, based upon the regulatory requirements for each site. Site-specific information, requirements and protocol are summarized in site-specific permits, and in attachments to this FOP.

Scope

This FOP is applicable to all Sandia Corporation (Sandia) employees and contractors who perform soil vapor monitoring activities at SNL/NM. Soil vapor monitoring is routinely performed at the Corrective Action Management Unit (CAMU) containment cell, Chemical Waste Landfill (CWL), Mixed Waste Landfill (MWL), Technical Area (TA)-V, and TA-III Classified Waste Landfill (CLWL). Site-specific information is provided in Attachments A, B, C, D, and E for the CAMU, CWL, MWL, CLWL, and TA-V, respectively. The general guidelines in this FOP may also be applied to non-routine soil vapor monitoring locations/events (*e.g.*, Tijeras Arroyo).

If other SNL/NM sites are subject to routine soil vapor monitoring in the future, additional attachments may be added to this FOP to address site-specific requirements and any variations to the general monitoring procedures covered in this FOP. If requirements change for the sites already addressed in this FOP, revisions will be made to the site-specific attachment.

Ownership

The Long Term Stewardship Department is responsible for development, approval, and revision of this document.

2.0 RESPONSIBLE INDIVIDUALS AND ORGANIZATIONS

The **Department Manager** is responsible for the following:

- Providing programmatic guidance leading to the development of this FOP.
- Review and approval of the procedure.
- Establishing and documenting field technician training in compliance with this FOP, site-specific permits (CAMU and CWL), and the MWL Long-Term Monitoring and Maintenance Plan (LTMMP).

The **Field Support Operations Project Lead** is responsible for the following:

- Coordinating with the Department Manager, Project Lead and Field Technicians regarding soil vapor sampling activities and the documentation of all required training.
- Assigning qualified Field Technicians to conduct the activities described in this procedure.
- Supervising the Field Technicians.
- Coordinating on-the-job (OJT) training for Field Technicians (trainees) performing the activities described in this procedure for the first time.
- Reviewing, implementing, and verifying the completion of all training required for Field Technicians.
- Providing Field Technicians with necessary equipment and supplies to conduct field work.
- Reviewing, revising, and maintaining technical work documents.

The **Project Lead** or designee is responsible for the following:

- Reviewing and concurring with this procedure and the related site-specific attachment(s).
- Providing overall coordination and management of site-specific soil vapor monitoring events.
- Providing copies of the relevant sections of the site-specific permit and sampling and analysis plan (SAP) (CAMU and CWL) and the MWL LTMMP for Field Technician review and signoff, prior to sampling.
- Reviewing field documentation and analytical results.
- Assisting with the revision of this procedure as necessary or every three years.

The **Field Technician** is responsible for the following:

- Completing all necessary and required training as specified by the Field Support Operations Project Lead. At a minimum, required training shall include the training defined in this FOP, site-specific permits (CAMU and CWL), and the MWL LTMMP.
- Maintaining requisite training status.
- Inspecting and maintaining equipment.
- Completing a tailgate safety briefing prior to each day's soil vapor monitoring activities.
- Collecting and storing samples properly, when applicable.
- Delivering samples to the Sample Management Office (SMO) in a timely manner, relative to analytical holding times, when applicable.
- Completing and reviewing field documentation forms.
- Inspecting soil vapor monitoring locations during each sampling event and documenting the inspections along with any deficiencies and/or repairs, or breach of monitoring location security. Reporting deficiencies and/or breach of security to the Field Support Operations Project Lead and the Project Lead.

- Providing recommendations for revisions to this procedure (*i.e.*, process improvement feedback as appropriate).

3.0 TRAINING QUALIFICATIONS

Personnel conducting soil vapor monitoring shall complete all training required to perform work under this FOP and in accordance with site-specific permits and the MWL LTMMP:

- Field personnel shall sign an Authorized Users List (EP2009-AUL) to affirm they have read and understand this document, and agree to operate within the stated constraints.
- Read SNL/NM Corporate Policy ESH100 Environment Safety & Health.
- Required department training and training identified in the primary hazard screening (PHS) results.
- Read applicable site-specific training (*i.e.*, PHS, health and safety plan, etc.)
- Read applicable sections of site-specific permits and SAPs (CAMU and CWL), the MWL LTMMP, and comply with the related training program requirements.
- Document site-specific permit training requirements (CAMU, CWL) for a Field Technician (on file at the CAMU Administrative Trailer).
- OJT, as necessary, for new personnel performing field activities. Document training by completing an OJT Form (EP 2009-OJT).
- Complete training courses listed in Table 1.

Table 1 - Training Course List

Course Code	Course Title
CHM100	Chemical Safety
CHM103	Site Specific Chemical Safety
ELC105	Basic Electrical Safety (> 50 volts)
ENV100	OSHA Health & Safety Basic Training – General Worker (40 HR)
ENV103	OSHA Health & Safety Training Refresher (8 HR)
ENV112	Hazardous Waste & Environmental Management Training
ESH100	Environment Safety& Health Awareness
MCH200	Hand and Power Tool Safety
MED102	Standard First Aid
MED104	Heartsaver CPR
OTS101	Occupational Thermal Stress
PPE106	Personal Protective Equipment Training
PRS150	Pressure Safety Orientation
PRS250	Advance Pressure Safety
RAD102	General Employee Radiological Training

4.0 HEALTH AND SAFETY

A task hazard analysis (THA) has been performed on the activities described in this FOP and is detailed in Section 4.1. The THA classifies the potential hazards and rates them based on the probability of occurrence. The THA lists control measures that will be used to mitigate the potential hazards. A site-specific PHS (see Section 9.0 for list of applicable PHSs) shall be completed prior to soil vapor monitoring activities to help identify potential hazards that can be expected when performing the work. The control measures may include exposure assessment surveys (by a SNL/NM industrial hygienist), courses, and training that are identified as part of the PHS results. This approach to identifying, rating, and controlling hazards is consistent with SNL/NM's Integrated Safety Management System initiative. Hazards classification is standard industrial hazards (SIH) for activities identified in this FOP.

A site-specific Activity Level Work Evaluation Form (EP 2009-ALW) shall be completed and approved by the Department Manager as required by administrative operating procedure (AOP) 09-10, *Work Planning and Controls*.

A site-specific tailgate safety and emergency response briefing shall be conducted by a qualified Field Technician each day before the start of field activities.

In the event that work is stopped due to:

- safety related issue(s),
- an injury incurred while performing the tasks identified in this procedure, or
- as the result of an audit,

the Field Technician shall immediately notify the Field Support Operations Team Lead, the Project Lead, and the Department Manager. The Field Technician shall seek the assistance of the Field Support Operations Team Lead for the mitigation of the hazard and the completion of a Work Resumption Authorization Form (EP 2009-WRA) as required by AOP 09-10, *Work Planning and Controls*. The Department Manager shall sign the completed form prior to the restart of work.

4.1 Task Hazard Analysis

Task Description - Soil Vapor Sampling for Volatile Organic Compounds

Soil vapor samples are collected from various SNL/NM sites (*e.g.*, CAMU, CWL, MWL, TA-V) and are analyzed to determine levels of volatile organic compound (VOC) contaminants in the surrounding soil pore space. VOC screening with a photoionization detector (PID) or equivalent detector is performed prior to sample collection to provide real-time data relative to stabilization of organic soil vapor concentrations during the purging process. (Note: Based upon historic soil

vapor concentrations documented at the sites, VOC screening with a PID or equivalent detector during the purging and sampling process is not necessary for worker health and safety purposes). The samples are collected by connecting tubing from a sampling pump to a sampling port on the soil vapor monitoring system. A SUMMA® canister is connected in line with the tubing system. The SUMMA® canister is under a vacuum and has a valve that when opened, draws in the vapor sample. The pump is run to purge the air from the sampling tube and draw representative soil vapor from the soil pore space surrounding the sampling port in the subsurface prior to collecting the soil vapor sample. After the air has been purged from the sampling tube and area immediately surrounding the sample port, the pump is turned off and a valve is opened on the SUMMA® canister which draws in the vapor sample. A THA is provided in Table 2.

Task Description – Soil Vapor Monitoring for Methane Gas

An instrument which is capable of measuring methane gas as a percentage of the lower explosive limit is used to monitor and report methane. An extension tube (rigid acrylic) with stopper is attached to the instrument and is lowered into monitoring ports. The stopper creates a seal that allows for *in situ* monitoring of methane gas that may have collected inside the port.

A THA for the activities soil monitoring for VOCs and methane gas is provided in Table 2.

Table 2 – Task Hazard Analysis
Soil Monitoring for VOCs and Methane Gas
Level of Protection—Level D Personal Protective Equipment (safety shoes/boots, safety glasses)

Potential Hazard	Hazard Rating	Control
Chemical (various VOCs)	SIH	• There will be no contact with contaminated soils during soil vapor monitoring activities. Soil vapors will be monitored using a PID as part of the purging process for VOC sampling. Historically VOC levels have been low (parts per million). Eating, drinking and smoking will not be permitted while performing soil monitoring activities.
Physical • Heat stress • Cold stress • Sunburn • Mechanical hazards • Pinch points • Strains, and lifting hazards • Slips, trips, falls • Motor vehicle accident • Electrical • Vacuum (negative pressure)	SIH	• Soil vapor monitoring activities are not physically demanding. Workers will be trained on heat stress, cold stress, and sunburn hazards. Sunscreen will be provided. • Appropriate inspections of equipment will be performed prior to use. • Leather work gloves will be worn when handling steel cable and removing vault covers. • Proper lifting techniques will be reinforced. • Proper housekeeping will be maintained. • Holes around monitoring area will be filled or covered to eliminate slip, trip hazards. • Seat belts will be worn anytime drivers and passengers are in a moving motor vehicle. • Proper ground fault circuit interrupter (GFCI) devices will be used for the electric equipment and tested before each use.

Table 2 – Task Hazard Analysis
Soil Monitoring for VOCs and Methane Gas (concluded)
Level of Protection—Level D Personal Protective Equipment (safety shoes/boots, safety glasses)

Potential Hazard	Hazard Rating	Control
Physical (concluded)	SIH	• A management approved pressure safety data package is in place for equipment used for soil vapor sampling.
Radiological	SIH	• There are no radiological hazards specifically related to soil vapor monitoring at the CAMU, CWL, MWL, CLWL, and TA-V.
Fire	SIH	• Fire extinguishers will be located in mobile equipment.

5.0 EQUIPMENT AND MATERIALS

5.1 Equipment and Materials for VOC Soil Vapor Sampling

The equipment and materials required for performing VOC soil vapor sampling are as follows:

- Analysis Request/Chain-of-Custody (AR/COC) forms and sample labels.*
- Logbook (if applicable).
- Field forms:
  - SUMMA® Canister Log (Attachment F).
  - Soil Vapor Sampling Log (Attachment G).
- AC power provided by ground fault circuit interrupter (GFCI) outlets.
- Vacuum pump and sampling manifold assembly.
- Flow rate meter.
- Vacuum gauge.
- VOC monitoring equipment (PID or equivalent).
- SUMMA® canister(s).
- Ultra-pure nitrogen gas cylinder for collecting quality control samples.
- Regulator manifold assembly specific to ultra-pure nitrogen quality control sample collection.
- Key(s) to unlock padlocks.

Additional equipment requirements may exist at the different sites. See Attachments A (CAMU), B (CWL), C (MWL), and D (TA-V) for site-specific requirements and protocol.

5.2 Equipment and Materials for Methane Gas Monitoring

The equipment and materials required for performing *in situ* soil vapor monitoring for methane gas are as follows:

- Logbook.

- A field form that meets program requirements.
- Calibrated instrument that measures percent of the lower explosive limit (LEL) for methane gas (obtain from the SNL/NM Instrumentation and Dosimetry Department).
- 48-inch extension sample tube with stopper for sealing sampling port.
- Watch.

Additional equipment requirements may exist at the different sites. See Attachment D (CLWL) for site-specific requirements and protocol.

6.0 PROCEDURES

6.1 SOIL VAPOR SAMPLING FOR VOCS

Note: Prior to conducting sampling refer to site-specific Attachments in this FOP for additional information.

Soil vapor sampling for VOCs involves pre-sampling preparation, monitoring system and equipment inspection, equipment set up and purging/sample collection, quality control sample collection (if required for site), and shipment of samples to the analytical laboratory. The following sections detail the overall soil vapor sampling procedure in the sequence the activities will be performed.

6.1.1 Pre-Sampling Preparations

The following must be completed before soil vapor sampling can begin:

1) Obtain the SUMMA® canisters from the SMO and check their vacuums by:
   - Connecting the vacuum gauge provided by the laboratory to the valve on top of the SUMMA® canister.
   - Open dial on needle valve.
   - Record vacuum for each canister on the SUMMA® Canister Log (Attachment F).
   - Close valve before removing vacuum gauge.

   The nominal vacuum at SNL/NM (approximate elevation 5,400 feet [ft]) is 23 to 25 inches (in.) mercury (Hg). A copy of the SUMMA® canister vacuum readings shall be sent to the laboratory with the canisters after soil vapor sampling is completed.
2) Obtain AR/COC and sample control numbers from the SMO Home Page, http://info.sandia.gov/esh/smo/index.html. Prepare and print out AR/COC and sample labels.
3) Calibrate the VOC monitoring instrument according to manufacturer's manual prior to use during sampling, or obtain monitoring instruments from the SNL/NM Safety and Health Instrumentation Program.

6.1.2 Equipment Setup and Sample Collection

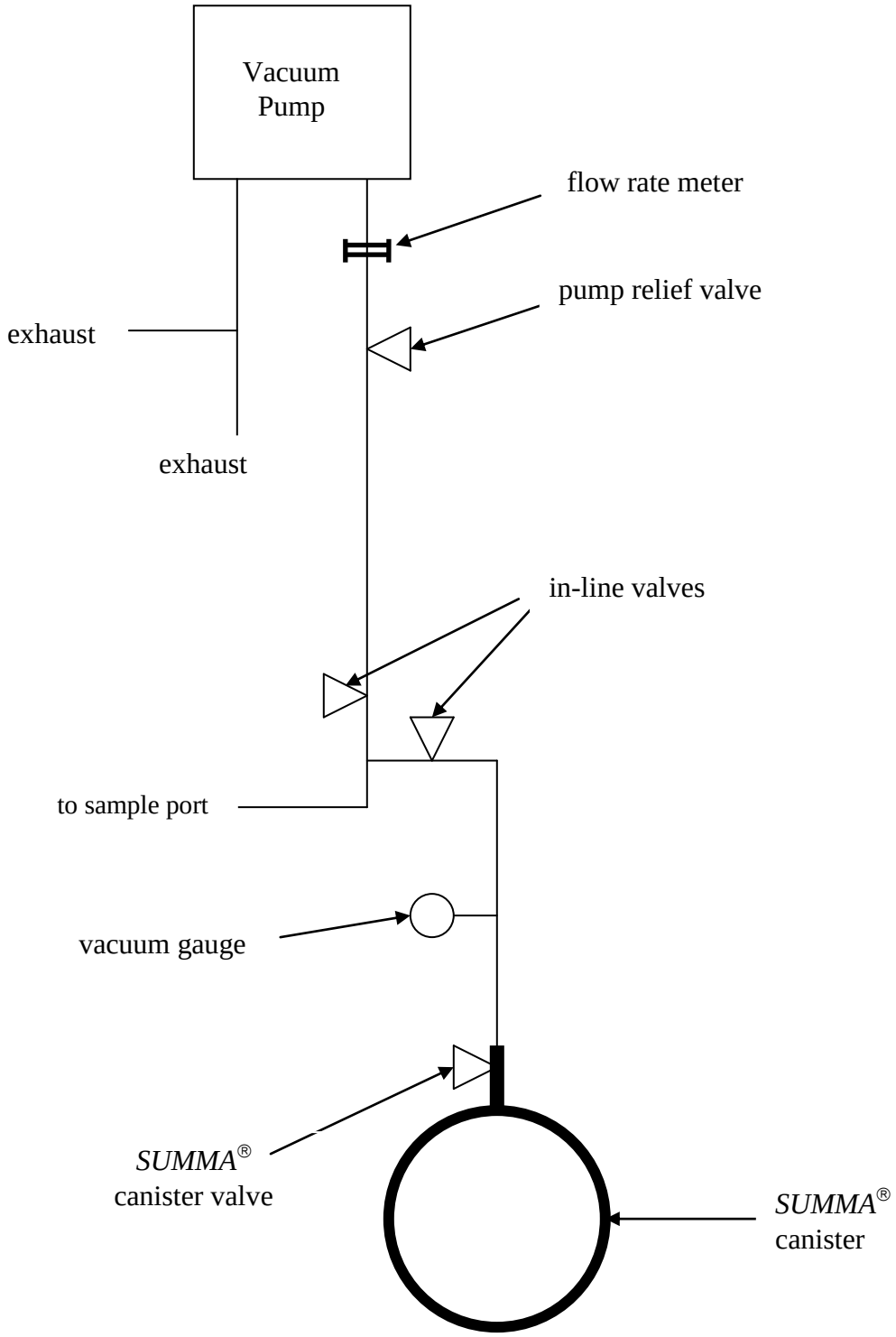
1) Load equipment detailed in Section 5.0 into sampling vehicle.
2) Position sampling vehicle adjacent to sampling location. The vehicle engine shall be turned off during purging and soil vapor sample collection.
3) Connect vacuum pump to AC power.

See Figure 6-3 for a general schematic of the vacuum pump and SUMMA® canister setup.

4) Connect stainless steel line from soil vapor sampling manifold to SUMMA® canister port.
5) Connect intake tube of vacuum pump to sampling port.
6) Open both in-line valves. Make sure SUMMA® canister valve and pump relief valve are closed.
7) Turn on pump to purge sampling tube and/or borehole. Use the sampling tube and/or borehole volume (see information below and Attachments A [CAMU], B [CWL], C [MWL], and D [TA-V] for site-specific purging information) and flow rate meter value to calculate the purge time (see purging information below).
8) Purge for length of time that allows a minimum of three volumes of the sampling tube and/or borehole to be purged (see purging information below).
9) After three volumes have been purged, monitor the VOC levels by attaching the VOC monitoring instrument to the exhaust port of the vacuum pump. Continue the purging process until the VOC levels stabilize. Record stabilized VOC reading on the Soil Vapor Sampling Log (Attachment G).
10) Upon completing purging of three sampling tube volumes and stable final VOC measurements, close in-line valve closest to the pump.
11) Open pump relief valve.
12) Open SUMMA® canister valve.
13) When the vacuum gauge on the manifold reaches approximately minus 10 in. Hg, close the SUMMA® canister valve. This will prevent the canister from going to ambient pressure (0 in. Hg).
    (Note: The analytical laboratory, Test America, requests that approximately minus 10 in. Hg of vacuum remain in the SUMMA® canister at completion of sampling.)
14) Remove manifold from the SUMMA® canister.
15) Verify end vacuum of approximately minus 10 in. Hg by connecting the vacuum gauge provided by the laboratory to the valve on top of the SUMMA® canister.
16) Open SUMMA® canister valve.
17) Record the ending vacuum for the canister on the SUMMA® Canister Log (Attachment F).
18) Close the SUMMA® canister valve, remove vacuum gauge and replace Swagelok® dust cap.

19) Disconnect intake tube of vacuum pump from sampling port and replace Swagelok® dust cap.
20) Fill out date and time on sample label and attach it to SUMMA® canister tag. Do not attach sample label to canister itself.
21) Complete appropriate Soil Vapor Sampling Log. Complete AR/COC.

Figure 6-3
Vacuum Pump and SUMMA® Setup



Purging Information

The purge time is a function of the volume of the sampling tube and/or borehole that needs to be purged and the flow rate through the sampling tube. A minimum of three sampling tube and/or borehole volumes are purged at each location before a sample is collected.

Volume calculations for cylindrical pipes and sampling tubes are as follows:

$V = \pi(D^2/4)L$ where: V = volume
D = diameter
L = length

Minimum pump running time to evacuate three sampling tube/well volumes from each sampling port is calculated as follows:

$t = (V/Q)*3$ where: t = time
V = volume
Q = flow rate

See Attachments A (CAMU), B (CWL), C (MWL), and D (TA-V) for site-specific purge volumes based upon individual soil vapor monitoring location construction details.

6.1.3 Quality Control Sample Equipment Setup and Sampling

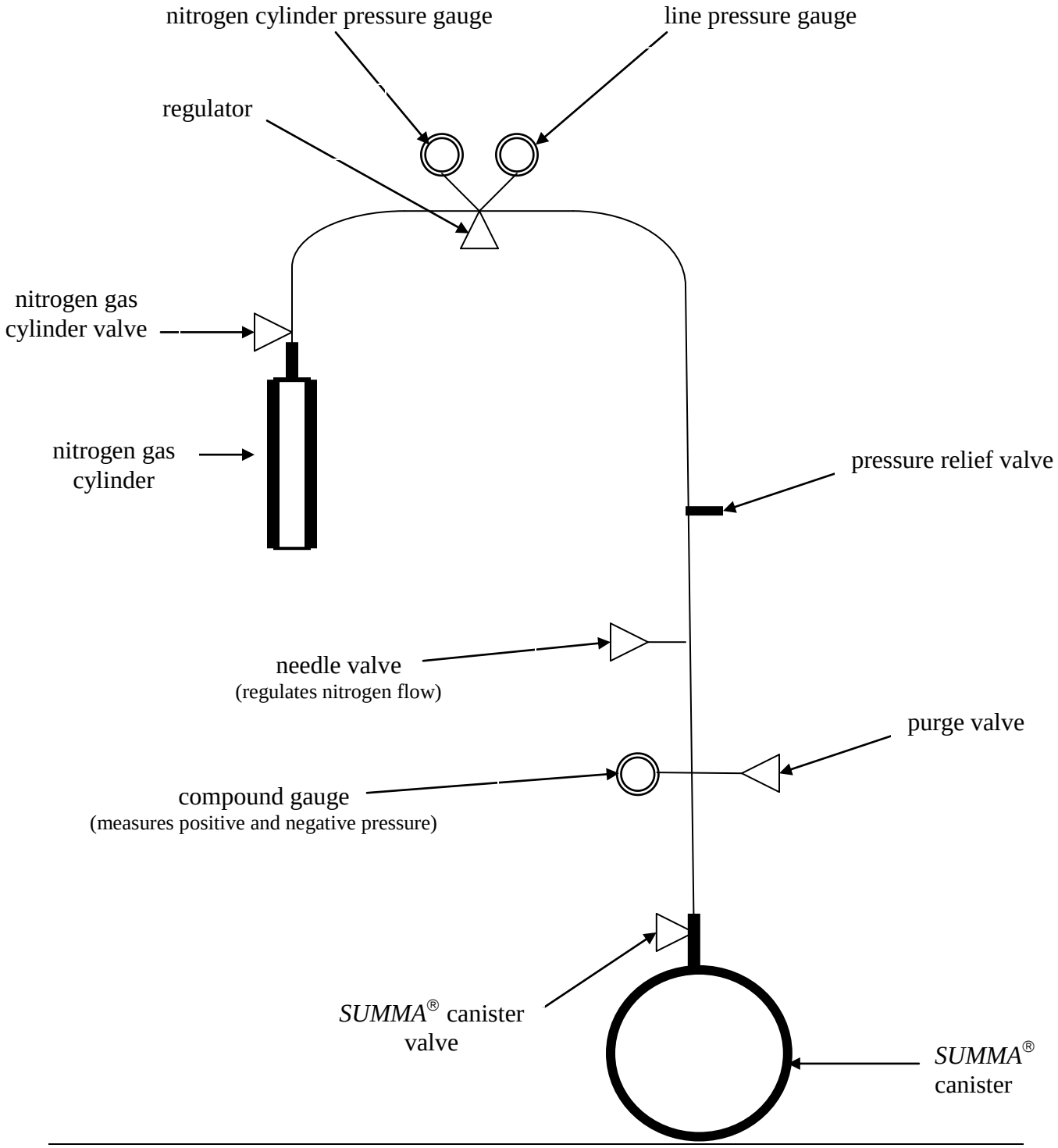
The site-specific SAP may require that a quality control sample of ultra-pure nitrogen gas be collected in a SUMMA® canister. The quality control sample shall be kept in the presence of the other SUMMA® canisters during routine sample collection and will accompany the routine samples to the laboratory for analysis.

Use the following procedure for collecting the ultra-pure nitrogen gas sample. See Figure 6-4 for diagram of equipment set up.

1) Close needle valve, purge valve, and regulator.
2) Connect regulator manifold assembly to SUMMA® canister and cylinder containing nitrogen gas.
3) Open nitrogen gas cylinder valve.
4) Adjust regulator to 8 pounds per square in. (psi) line pressure.
5) Adjust needle valve until compound gauge measures positive 8 psi.
6) Close nitrogen gas cylinder valve.
7) Open purge valve to purge line.
8) Close purge valve when compound gauge measures zero.
9) Repeat steps 3 through 8 a total of two times.
10) Open nitrogen gas cylinder valve.

11) Open SUMMA® canister valve.
12) Close SUMMA® canister valve when compound gauge measures negative 10 in. of Hg.
13) Close nitrogen gas cylinder valve.
14) Open purge valve.
15) Disconnect regulator manifold assembly from SUMMA® canister and nitrogen gas cylinder.
16) Close needle valve, purge valve, and regulator.

Figure 6-4
Quality Control Sample Regulator Manifold and SUMMA® Setup



6.1.4 Shipping Samples to Laboratory

The SUMMA® canisters, AR/COC, and SUMMA® Canister Log will be taken to the SMO for shipment to the laboratory.

6.2 METHANE GAS MONITORING

1) Attach 48-inch extension tube with stopper to the methane gas monitoring instrument. The stopper that seals the sampling port should be located 30 inches from the open end of the extension tube.
2) Turn on methane gas monitoring instrument.
3) Remove end cap from monitoring port.
4) Immediately lower extension tube into monitoring port with the stopper creating a seal at the top of the sampling port.
5) After 2 minutes, record methane gas measurement and any other applicable gas values.
6) Remove extension tube.
7) Reseal monitoring port with end cap.

6.3 INSPECTIONS

Inspections of soil vapor monitoring locations and equipment shall be performed in accordance with site requirements (*i.e.*, permits, MWL LTMMP). An example of an inspection form is provided in Attachment H. (Note: Inspection frequency and the format of inspection forms will vary based on site-specific requirements detailed in applicable Permits or regulatory documents.) Deficiencies and repairs shall be documented per site requirements.

7.0 QUALITY ASSURANCE / QUALITY CONTROL

See site-specific quality assurance/quality control (QA/QC) guidelines and requirements as detailed in Attachments A through E and associated site-specific permits (CAMU and CWL) and the MWL LTMMP.

8.0 RECORDS

Analytical reports will be provided with acceptable QA/QC. The following records will be maintained at the Customer Funded Record Center:

- authorized user list
- sampling and analytical results
- field forms
- inspection forms
- logbooks (if applicable).

Sampling results shall be kept electronically in the Environmental Data Management System database. Copies of logbooks (if applicable), authorized user list, field and inspection forms shall be maintained at the CAMU Administrative Trailer for the CAMU, CWL, and MWL per site-specific permits (CAMU and CWL) and the MWL LTMMP. Training records shall be kept electronically in the Training and Employee Development System (TEDS) database. TEDS shall be accessible from the CAMU Administrative Trailer. Copies of inspection forms for the CAMU, CWL, and MWL shall be included in annual reports.

9.0 REFERENCES

Appendix E of the Class III Permit Modification for the Management of Hazardous Remediation Waste in the CAMU, Technical Area III, SNL/NM, ER Project, September 1997, Final, as amended.

ASSOP 01-04, "ASSOP Active Soil-Gas Sampling Using Method TO-14 at the CAMU", SNL/NM, November 2001.

Environment Safety & Health Manual, SNL/NM, (latest edition).

New Mexico Environment Department (NMED), October 2009. "Final Permit Decision and Response to Comments, Post-Closure Care Permit for the Chemical Waste Landfill, Sandia National Laboratories, EPA ID# NM5890110518, SNL-06-002." New Mexico Environment Department Hazardous Waste Bureau, Santa Fe, New Mexico.

New Mexico Environment Department (NMED), March 2012. "New Mexico Solid Waste Rules, Solid Waste Management Act, Article 8 and Article 9, Solid Waste Rules 20.9.2 – 20.9.10 NMAC". New Mexico Environment Department Solid Waste Bureau, Santa Fe, New Mexico.

PLA 04-01, "Health and Safety Plan for the CAMU Containment Cell", SNL/NM, Environmental Programs and Assurance, (latest edition).

SNL PHS # SNL05A01119 "CAMU Containment Cell Monitoring", SNL/NM, (latest edition).

SNL PHS # SNL06A00497 "Vadose Zone Monitoring at the Mixed Waste Landfill", SNL/NM, (latest edition).

SNL PHS # SNL11A00081 "Environmental Programs Soil Vapor Well Sampling", SNL/NM (latest edition).

SNL PHS # SNL13A00003 "TAIII Classified Waste Landfill Quarterly Methane Monitoring", SNL/NM (latest edition).

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Attachment A

Corrective Action Management Unit

Site-Specific Information

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Corrective Action Management Unit (CAMU) Introduction and Background

Soil vapor monitoring requirements are defined in Appendix E (Proposed Alternative to Groundwater Monitoring for the Corrective Action Management Unit) of the CAMU Permit Application (SNL/NM September 1997), incorporated by reference as part of the Hazardous and Solid Waste Amendments Module of the Resource Conservation and Recovery Act (RCRA) Permit issued by U.S. Environmental Protection Agency (EPA) Region 6 (EPA 1993) and administered by the New Mexico Environment Department (NMED).

Prior to performing work field technicians shall complete/document all required training as indicated in Table 1 of FOP 08-22, *Soil Vapor Sampling* and pertinent training as listed in PLA 04-01, *Health & Safety Plan for the Corrective Action Management Unit*.

CAMU Soil Vapor Sampling Network

The CAMU uses the following three monitoring subsystems to monitor for volatile organic compounds (VOCs) as supplemental data for the CAMU Vadose Zone Monitoring System (VZMS) leak detection program:

VSA - The Vertical Sensor Array (VSA) consists of eleven pairs of vertically oriented monitoring locations. Five are located on both the eastern and western margins of the containment cell. The eleventh monitoring location is situated at the northern end of the cell. Each VSA location contains a soil vapor sampling port at 5 ft and 15 ft beneath the containment cell sub-liner. Tubing extends from the two soil vapor sampling ports and terminates in the above ground enclosure (AGE) where there are connections for the sampling tube to connect to the vacuum pump.

CSS - The six Chemical Waste Landfill Sanitary Sewer (CSS) boreholes are located between the CAMU containment cell and the sanitary sewer line. Each monitoring location consists of a 2 in. diameter steel pipe driven to approximately 20 feet (ft) below grade. Each pipe has a screened section at the bottom to allow for soil vapor sampling. 2 ft of the pipe protrudes above ground and is protected by a steel casing with a locking cap.

PSL - The Primary Sub-Liner (PSL) consists of five, 6-inch (in.) inside diameter vitrified clay pipe (VCP) runs that are oriented horizontally under the CAMU containment cell. The end of each VCP run is connected to 6 in. poly vinyl chloride (PVC) pipe risers that are located on the north and south ends of the CAMU containment cell. The PVC risers are protected above ground by locked steel casings. The PVC risers are used to access the PSL system.

CAMU Equipment Setup and Sampling Process

Follow the procedure detailed in Section 6.3 of FOP 08-22 and the specific instructions provided below for the VSA, CSS, and PSL subsystems respectively.

VSA Subsystem

1) Position sampling vehicle adjacent to VSA location's AGE.
2) Unlock and remove padlock from AGE.
3) Release the two door clamps and open the door.
4) The 5-ft and 15-ft soil vapor sample ports are clearly labeled inside AGE. Remove Swagelok® dust cap and connect intake tube from vacuum pump to the appropriate soil vapor sampling port (5-ft or 15-ft).
5) Collect soil vapor sample as described in Section 6.3 of FOP 08-22.
6) After samples have been collected, replace Swagelok® dust cap, close door and secure door clamps. Lock padlock on AGE hasp.
7) Repeat steps 2 through 6 for remaining VSA locations.

CSS Subsystem

1) Position sampling vehicle adjacent to CSS well to be sampled.
2) Unlock and open wellhead lid.
3) Remove Swagelok® dust cap from the soil vapor sample port on top of the well standpipe.
4) Collect soil vapor sample as described in Section 6.3 of FOP 08-22.
5) After soil vapor sample has been collected, replace Swagelok® dust cap and close and lock wellhead lid.
6) Repeat steps 2 through 5 for remaining CSS boreholes.

PSL Subsystem

Equipment items required in addition to those listed in Section 5.0 of FOP 08-22 include:

1) Aluminum centralizer.
2) Pulley assembly with locking pin.
3) Cable guide.
4) Cable winch (mounted to floor of sampling vehicle).
5) 500 ft polyethylene tube on a reel. The tube has a 0.25 in. outside diameter and 0.17 in. inside diameter.
6) Two-way radios.

At the south end of the CAMU containment cell:

1) Position vehicle so cable winch is aligned with south opening of PSL to be sampled.
2) Thread winch cable and polyethylene tube through pulley assembly.
3) Loosen allen screws on slotted end of aluminum capsule/centralizer.
4) Remove slotted end cap and feed winch cable through slot.
5) Reattach slotted end cap and secure allen screws.
6) Attach polyethylene tube from reel to aluminum capsule/centralizer using duct tape.
7) Unlock and open protective steel casing cap.
8) Unhook resident steel cable from 6 in. PVC cap and attach resident steel cable to swivel/carabiner assembly on aluminum capsule/centralizer.
9) Place aluminum capsule/centralizer into opening of PVC pipe.
10) Slide pulley assembly into unistrut fixture mounted on top inside of protective steel casing. Use locking pin to hold pulley assembly in place.
11) Take up slack in winch cable and zero cable winch footage counter.
12) Release winch drive lock to allow winch drum to turn freely.
13) Remain with vehicle to monitor cable winch while coworker goes to north end of PSL.

At the PSL north end:

14) Unlock protective steel casing cap and remove 6 in. PVC cap.
15) Unhook resident steel cable end from 6 in. PVC cap and thread resident steel cable through cable guide. Immediately hook resident steel cable end back to 6 in. PVC cap to prevent cable end from sliding into PSL.
16) Insert cable guide into unistrut fixture mounted on inside bottom of protective steel casing.
17) Notify coworker on south end by two-way radio and begin pulling resident steel cable. Pull resident steel cable 170 ft to position gas sampling tube in midway point of PSL.

At the PSL south end:

18) Polyethylene tube from reel and winch cable are played out simultaneously while resident steel cable is pulled through PSL.
19) South end worker will use 2-way radio to notify north end worker when footage counter indicates polyethylene tube is positioned 170 ft inside PSL.
20) Connect intake tube of vacuum pump to the Swagelok® connector on polyethylene tube at reel hub.
21) Collect sample as described in Section 6.3 of FOP 08-22.
22) After soil vapor sample has been collected, disconnect intake tube of vacuum pump from polyethylene tube at reel hub. Retrieve winch cable and 170 ft polyethylene tube simultaneously.
23) Disconnect resident steel cable from aluminum capsule/centralizer and reattach it to 6 in. PVC cap.

24) Replace 6 in. PVC end cap and lock protective steel casing cap.

At the PSL north end:

25) Unhook resident steel cable from 6 in. PVC cap and remove cable guide.
26) Reattach resident steel cable to 6 in. PVC cap.
27) Replace 6 in. PVC end cap and lock protective steel casing cap.

Repeat steps 7 through 27 for the remaining PSL locations.

CAMU Tube Volumes and Purge Time Calculations

Standard practice calls for purging soil vapor until a minimum of three tube volumes are evacuated. Volume calculations and purge time are calculated below. Because minimum purge times are so small, they have been increased to a required purge time. Table A-1 presents the purge volumes, minimum purge times, and the required purge times for the VSA, CSS, and PSL soil vapor monitoring locations.

Volume calculations for tubes are as follows:

$V = \pi(D^2/4)L$ where: V = volume
D = diameter
L = length

Minimum pump run time to evacuate three tube volumes is calculated as follows:

$t = (V/Q)*3$ where: t = time
V = volume
Q = flow rate (has been predetermined for the VSA, CSS, and PSL)

VSA Subsystem

Volume of the VSA soil vapor screen and ¼-inch polyethylene sampling tube is calculated as follows:

V of soil vapor screen $= \pi * [(2\text{ in.})^2 / 4] * 12\text{ in.} * [1\text{ft}^3 / (12\text{ in.})^3] = 0.022\text{ ft}^3$
V of sampling tube $= \pi * [(0.25\text{ in.})^2 / 4] * 50\text{ ft} * (12\text{ in./ft}) * [1\text{ft}^3 / (12\text{ in.})^3] = 0.017\text{ ft}^3$

Minimum pump running time to evacuate three volumes from each VSA sampling port is calculated as follows:

$t = [(0.022\text{ ft}^3 + 0.017\text{ ft}^3) / (1.3\text{ ft}^3/\text{minute})] * 3 = 0.09$ minute or 5 seconds

CSS Subsystem

Volume of the CSS tube (galvanized well pipe) are calculated as follows:

V of galvanized pipe = π * [(2.07 in.)^2 / 4] * 21 ft * (12 in./ft) * [1ft^3 / (12 in.)^3] = 0.49 ft^3

Minimum pump running time to evacuate three volumes from each CSS well is calculated as follows:

t = [(0.49 ft^3) / (1.3 ft^3/minute)] * 3 = 1.13 minutes or 68 seconds

PSL Subsystem

Based on the construction of the PSL monitoring subsystem, it is not practical to purge the large tube volumes (VCP + PVC) associated with each of the five monitoring locations. Instead, the purge volume and purge time is based on the length of sampling tube inserted into each of the PSL tubes.

The soil vapor samples drawn from the VCPs in the PSL are taken from midway down the length of each pipe. 500 ft of polyethylene tube, with a 0.17 in. nominal inside diameter, is unrolled from a reel and pulled down the VCPs with a winch and wire cable to the midpoint of the VCPs. The other end of the tube is connected to the soil vapor sampling system. The vacuum pump is used to draw soil gas from the VCPs and evacuate the 500 ft of polyethylene tube.

V of sampling tube = π * [(0.17 in.)^2 / 4] * 500 ft * (12 in./ft) * [1ft^3 / (12 in.)^3] = 0.079 ft^3

Minimum pump running time to evacuate three volumes from the 500 ft. reel of tube is calculated as follows:

t = [(0.079 ft^3) / (1.3 ft^3/minutes)] * 3 = 0.18 minute or 11 seconds

Table A-1 CAMU VZMS Soil Vapor Sampling Purge Volumes and Purge Time

System	VSA	CSS	PSL
Purge Volume (ft^3)	0.039	0.49	0.079
Minimum Purge Time (seconds)	5	68	11
Required Purge Time (minutes)	2	3	5

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Attachment B

Chemical Waste Landfill

Site-Specific Information

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Chemical Waste Landfill (CWL) Introduction and Background

Soil vapor sampling at the CWL shall be performed under the New Mexico Environment Department (NMED) approved Post-Closure Care Permit (PCCP) (NMED October 2009 and subsequent revisions). The PCCP includes a description of the soil gas monitoring process and network, as well as a Soil Gas Sampling and Analysis Plan (SAP). The SAP also references Sandia National Laboratories/New Mexico (SNL/NM) operating procedures and the *Sample Management Office Statement of Work for Analytical Laboratories and Quality Assurance Project Plan*. In all cases, the requirements of the PCCP SAP take precedence over those of any other referenced or listed document and/or procedure, including FOP 08-22, *Soil Vapor Sampling*.

Prior to performing CWL soil vapor sampling, field technician must meet all training requirements as specified in the PCCP.

CWL Soil Vapor Sampling Network

The CWL soil vapor sampling network consists of the following five soil vapor monitoring wells: UI-1, UI-2, D-1, D-2, and D-3. The UI designation refers to "Upper Intermediate" indicating the general depth horizon that these wells are designed to sample. The D designation refers to "Deep" and is similarly indicative of the sampling depth interval. There are three soil vapor sampling ports associated with each of the UI series wells and five soil vapor sampling ports associated with each of the D series wells. One soil vapor screen at each sampling depth consists of a 2 ft long by 0.31 in. inner diameter stainless steel screen that is attached to a 0.215 in. stainless steel tube that extends to the surface.

CWL Equipment Setup and Sampling Process

Follow the applicable sections of FOP 08-22, *Soil Vapor Monitoring*, and Attachment 3 of the CWL PCCP.

CWL Sampling Tube Volume and Purge Time Calculations

V of soil vapor screen = π * [(0.31 in.)^2 / 4] * 2 ft * (12 in./ft) * [1ft^3 / (12 in.)^3]= 0.0010 ft^3
V of sampling tube = π * [(0.215 in.)^2 / 4] * tube length (ft) * (12 in./ft) * [1ft^3 / (12 in.)^3]
Vapor Well Volume = (V soil vapor screen + V of sampling tube)
V to purge = 3 * (Vapor Well Volume)

The sampling locations, associated ports, corresponding sampling depths, and sampling tube volumes are presented in Table B-2.

Table B-2 CWL Purge Volume Calculations

Sampling Location	Port #	Soil Vapor Screen Volume (ft^3)	Sampling Tube Length = sample depth (ft. bgs) + riser (ft)	Sampling Tube Volume (ft^3)	Vapor Well Volume (ft^3)	Volume to purge (ft^3)
UI-1	1	0.0010	120+4.8 = 124.8	0.0315	0.033	0.098
	2	0.0010	80+4.8 = 84.8	0.0214	0.022	0.067
	3	0.0010	40+4.8 = 44.8	0.0113	0.012	0.037
UI-2	1	0.0010	136+3.1 = 139.1	0.0351	0.036	0.108
	2	0.0010	76+3.1 = 79.1	0.0199	0.021	0.063
	3	0.0010	36+3.1 = 39.1	0.0099	0.011	0.033
D-1	1	0.0010	470+4=474	0.1194	0.120	0.362
	2	0.0010	350+4=354	0.0892	0.090	0.271
	3	0.0010	240+4=244	0.0615	0.063	0.188
	4	0.0010	160+4=164	0.0413	0.042	0.127
	5	0.0010	100+4=104	0.0262	0.027	0.082
D-2	1	0.0010	470+1.7 = 471.7	0.1189	0.120	0.360
	2	0.0010	440+1.7 = 441.7	0.1113	0.112	0.337
	3	0.0010	350+1.7 = 351.7	0.0886	0.090	0.269
	4	0.0010	240+1.7 = 241.7	0.0609	0.062	0.186
	5	0.0010	120+1.7 = 121.7	0.0307	0.032	0.095
D-3	1	0.0010	480+3 = 483	0.1217	0.123	0.368
	2	0.0010	440+3 = 443	0.1116	0.113	0.338
	3	0.0010	350+3 = 353	0.0890	0.090	0.270
	4	0.0010	170+3 = 173	0.0436	0.046	0.134
	5	0.0010	120+3 = 123	0.0310	0.032	0.096

Minimum pump run time to evacuate three volumes from each sampling port is calculated as follows:

t = V to purge / Q where: t = time
V = volume
Q = flow rate (to be determined in the field based on equipment limitations)

Attachment C

Mixed Waste Landfill

Site-Specific Information

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Mixed Waste Landfill Introduction and Status

Soil vapor sampling at the MWL shall be performed under the New Mexico Environment Department (NMED) approved LTMMP (NMED January 2014). The LTMMP includes a description of the soil vapor monitoring process and network, as well as a Soil-Vapor Sampling and Analysis Plan (SAP). The SAP also references Sandia National Laboratories/New Mexico (SNL/NM) operating procedures and the *Sample Management Office Statement of Work for Analytical Laboratories and Quality Assurance Project Plan*. In all cases, the requirements of the LTMMP SAP take precedence over those of any other referenced or listed document and/or procedure, including FOP 08-22, *Soil Vapor Sampling*.

Prior to performing soil vapor sampling at the MWL, field technicians shall read the pertinent sections of the LTMMP.

MWL Soil Vapor Sampling Network

The MWL soil vapor sampling network consists of the following five soil vapor monitoring wells:

- MWL-SV-01
- MWL-SV-02
- MWL-SV-03
- MWL-SV-04
- MWL-SV-05

The soil vapor implant at MWL-SV-01 and MWL-SV-02 consists of a 0.5 ft long by 0.5 in. diameter stainless steel screen. It is attached to a nominal 0.25 in. diameter polyethylene tube that extends 41 ft to the ground surface and a sampling port.

The soil vapor sampling systems at MWL-SV-03, MWL-SV-04, and MWL-SV-05 consists of three Flexible Liner Underground Technologies (FLUTe^{TM}) multi-port soil-vapor monitoring wells with five sampling ports per location. Sampling ports are set at 50, 100, 200, 300, and 400 foot depths and attach to nominal 0.25 in. diameter polyethylene tubing. Each sampling interval spans a 5 ft depth interval. [Note: Installation of these three soil vapor monitoring wells is scheduled for May-June 2014. The NMED-approved Installation Plan requires sampling ports to be installed within "….plus or minus 10 vertical feet of the planned depth." Minor adjustments to the sampling and purging process based on actual as-built specifications will be incorporated as appropriate and documented in required MWL LTMMP annual reports.]

MWL Equipment Setup and Sampling Process

Follow the applicable sections FOP 08-22, *Soil Vapor Monitoring*, and any applicable information in the approved version of the LTMMP.

MWL Sampling Tube Volume and Purge Time Calculations

Calculations for MWL-SV-01 & MWL-SV-02:

V of soil vapor implant = π * [(0.5 in.)^2 / 4] * 0.5 ft * (12 in./ft) * [1ft^3 / (12 in.)^3]= 0.0007 ft^3
V of sampling tube = π * [(0.25 in.)^2 / 4] * 41 ft * (12 in./ft) * [1ft^3 / (12 in.)^3] = 0.0140 ft^3
Vapor Well Volume = (V soil vapor screen + V of tubing)
V to purge = 3 * (Vapor Well Volume)

Calculations for MWL-SV-03, MWL-SV-04, MWL-SV-05:

V of soil vapor implant = π * [(0.25 in.)^2 / 4] * 5 ft * (12 in./ft) * [1ft^3 / (12 in.)^3] = 0.0017 ft^3
V of sampling tube = π * [(0.25 in.)^2 / 4] * 50 ft * (12 in./ft) * [1ft^3 / (12 in.)^3] = 0.0170 ft^3
V of sampling tube = π * [(0.25 in.)^2 / 4] * 100 ft * (12 in./ft) * [1ft^3 / (12 in.)^3] = 0.0341 ft^3
V of sampling tube = π * [(0.25 in.)^2 / 4] * 200 ft * (12 in./ft) * [1ft^3 / (12 in.)^3] = 0.0681 ft^3
V of sampling tube = π * [(0.25 in.)^2 / 4] * 300 ft * (12 in./ft) * [1ft^3 / (12 in.)^3] = 0.1022 ft^3
V of sampling tube = π * [(0.25 in.)^2 / 4] * 400 ft * (12 in./ft) * [1ft^3 / (12 in.)^3] = 0.1363 ft^3
Vapor Well Volume = (V soil vapor screen + V of tubing)
V to purge = 3 * (Vapor Well Volume)

The sampling locations, associated ports, corresponding sampling depths, and purge volumes are presented in Table C-1.

Table C-2 MWL Purge Volume Calculations

Sampling Locations	Port #	Soil Vapor Implant Volume (ft^3)	Sample Depth (bgs) tubing length (ft)	Sampling Tube Volume (ft^3)	Vapor Well Volume (ft^3)	Volume to purge (ft^3)
MWL-SV-01 MWL-SV-02	1	0.0007	41	0.0140	0.015	0.044
MWL-SV-03 MWL-SV-04 MWL-SV-05	1	0.0017	50	0.0170	0.019	0.057
	2		100	0.0341	0.036	0.090
	3		200	0.0681	0.070	0.210
	4		300	0.1022	0.104	0.312
	5		400	0.1363	0.138	0.414

Minimum pump running time to evacuate three volumes from each sampling port is calculated as follows:

t = V to purge / Q where: t = time
V = volume
Q = flow rate (to be determined in the field based on equipment limitations)

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Attachment D

Technical Area III Classified Waste Landfill

Site-Specific Information

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Technical Area III (TA-III) Classified Waste Landfill Introduction and Background

In situ monitoring for methane gas is performed quarterly at the TA-III Classified Waste Landfill (CLWL) in accordance with New Mexico Administrative Code (NMAC), Title 20 (Environmental Protection), Chapter 9 (Solid Waste), Part 5 (Solid Waste Facility and Registered Facility Operating Requirements), Section C. An instrument which is capable of measuring methane gas as a percentage of the lower explosive limit is used to monitor and report methane gas concentrations. Other compounds that are measured include percent oxygen, carbon monoxide (parts per million [ppm]), and hydrogen sulfide (ppm).

CLWL Methane Gas Monitoring Network

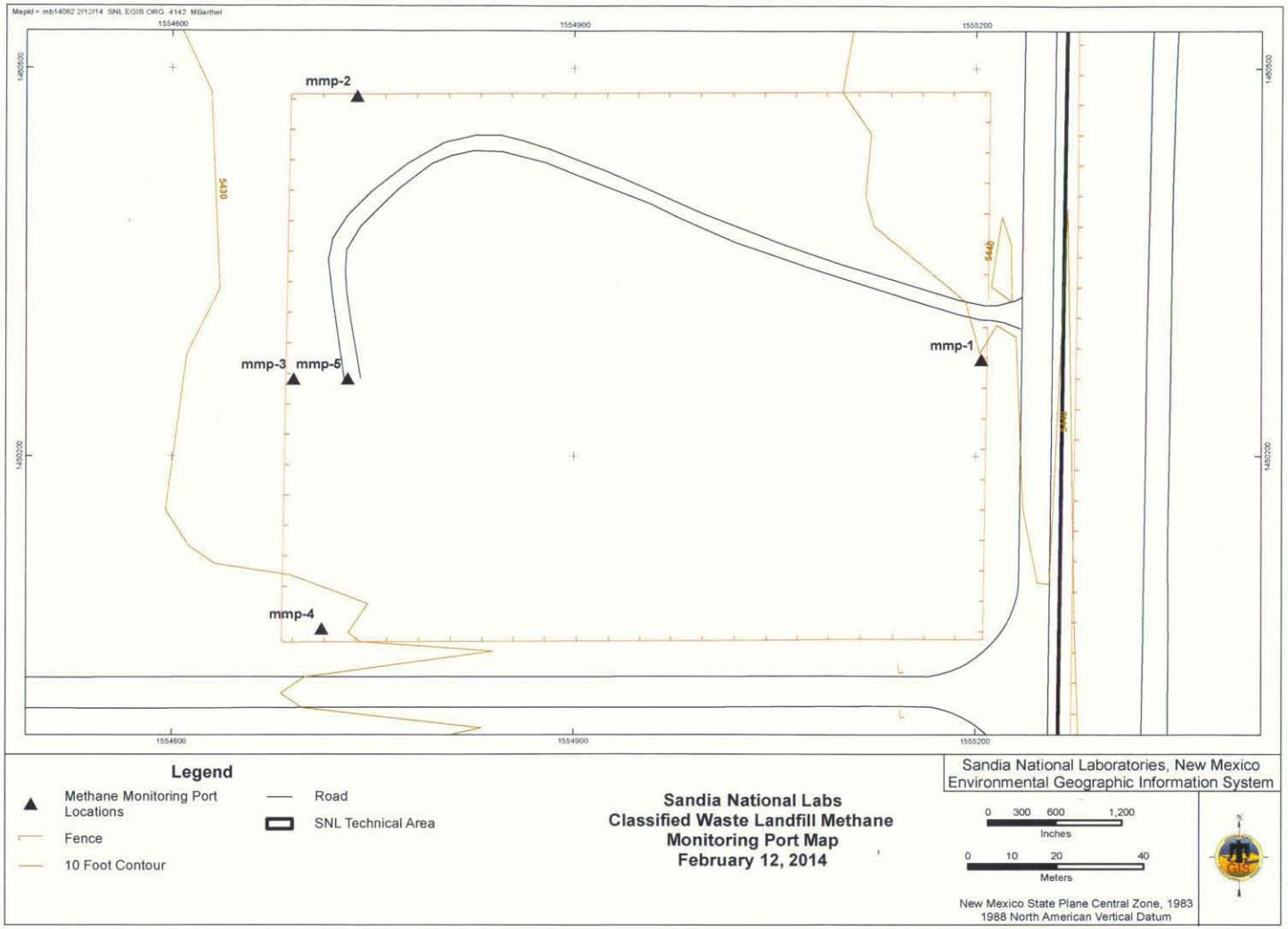
The CLWL methane gas monitoring network (Figure 1) consists of five methane monitoring port (MMP) locations. MMP-1 is located on the east side of the site approximately 3 feet from the site perimeter fence. MMP-2, 3, and 4 are located near the only trench containing waste and within 3 feet of the site perimeter fence. MMP-5 is located immediately east of the waste containing trench.

Each port consists of a 48-inch long, ¾-inch outer diameter, open ended, galvanized steel pipe, installed in a slightly larger diameter pilot hole approximately 36 inches deep, leaving 12-inches of the pipe exposed above ground surface. Loose soil was used to backfill the open space around each pipe. Each pipe has three columns of 1/8-inch diameter holes, four per column. The columns are spaced approximately 120 degrees apart radially. The holes start 3-inches from the bottom of the open ended pipe and are spaced vertically 1-inch apart. A threaded end cap is used to seal the section of pipe exposed at the surface. The end cap is removed when monitoring is performed. The attached methane monitoring field form is used when performing methane gas monitoring at the CLWL.

CLWL Equipment Setup and Monitoring Process

Follow the applicable sections of FOP 08-22, *Soil Vapor Monitoring*. Use the attached monitoring form and record values for all specified compounds.

Figure 1. TA-III Classified Waste Landfill Monitoring Locations Map



SANDIA NATIONAL LABORATORIES/NM
TECH AREA III CLASSIFIED WASTE LANDFILL
METHANE MONITORING FORM

Date:_____ Start time: _____ End time: _____

Barometric Pressure: _____ millibars Temperature: _____° C

Weather conditions:_____ Wind speed: _____ Wind direction: _____

Date and amount of last precipitation (within last 48 hours): _____

Instrument: Industrial Scientific ITX Multi-Gas Monitor (O_2/LEL/CO/H_2S)

Calibration expiration date: _____

Methane monitoring type: Dedicated probe port Probe depth: ~30 inches

Table of Results

Port Location, see attached map	Methane, % LEL	Oxygen, %	Carbon Monoxide, parts per million	Hydrogen Sulfide, parts per million
Port 1				
Port 2				
Port 3				
Port 4				
Port 5				

Sampler: _____

Signature: _____

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Attachment E

Technical Area V

Site-Specific Information

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Technical Area V (TA-V) Introduction and Background

In April 2004, the New Mexico Environment Department (NMED) issued a Compliance Order on Consent (NMED April 2004) to the U.S. Department of Energy (DOE) and Sandia Corporation, that identified TA-V as an area of groundwater contamination at Sandia National Laboratories/New Mexico (SNL/NM) requiring completion of a Corrective Measures Evaluation (CME). A CME Work Plan was prepared and implemented and a CME Report was submitted by SNL/NM in July 2005. In July 2008, the NMED issued the first Notice of Disapproval (NOD) to the DOE and Sandia regarding the CME Report for the TA-V study area at SNL/NM, which required further characterization of groundwater and soil vapor at TA-V. SNL/NM prepared the *Technical Area V Groundwater Investigation Work Plan* and submitted the plan to the NMED in April 2009. SNL/NM received NODs from NMED in August 2009 and December 2009, and submitted revised work plans in November 2009 and February 2010. The NMED conditionally approved the SNL/NM February 2010 *Technical Area V Groundwater Investigation Work Plan* in May 2010.

The approved work plan included the design of the soil vapor monitoring system required to provide data regarding vadose-zone volatile organic compound (VOC) profiles with depth. The work plan also discussed soil vapor sampling field activities such as preparation, purging, VOC monitoring, sample collection, and sample shipping. As established in the regulatory-approved work plan, requirements for soil sampling are addressed by the procedures documented in FOP 08-22, *Soil Vapor Sampling*. Information on the monitoring network and soil vapor sampling specific to TA-V (necessary to fulfill the requirements of the work plan) are provided below.

TA-V Soil Vapor Sampling Network

The TA-V soil vapor sampling network consists of three soil vapor monitoring wells (TAV-SV01, TAV-SV02, and TAV-SV03), with soil vapor sampling ports at depths of approximately 50 feet (ft), 100 ft, 150 ft, 200 ft, 250 ft, 300 ft, 350 ft, 400 ft, 450 ft, and 500 ft below ground surface (bgs). The soil vapor screen at each location consists of a 1-ft long by 0.5-in. diameter stainless steel screen. It is attached to 0.25 in. outside diameter stainless steel tube that extends to the ground surface and a sampling port.

TA-V Equipment Setup and Sampling Process

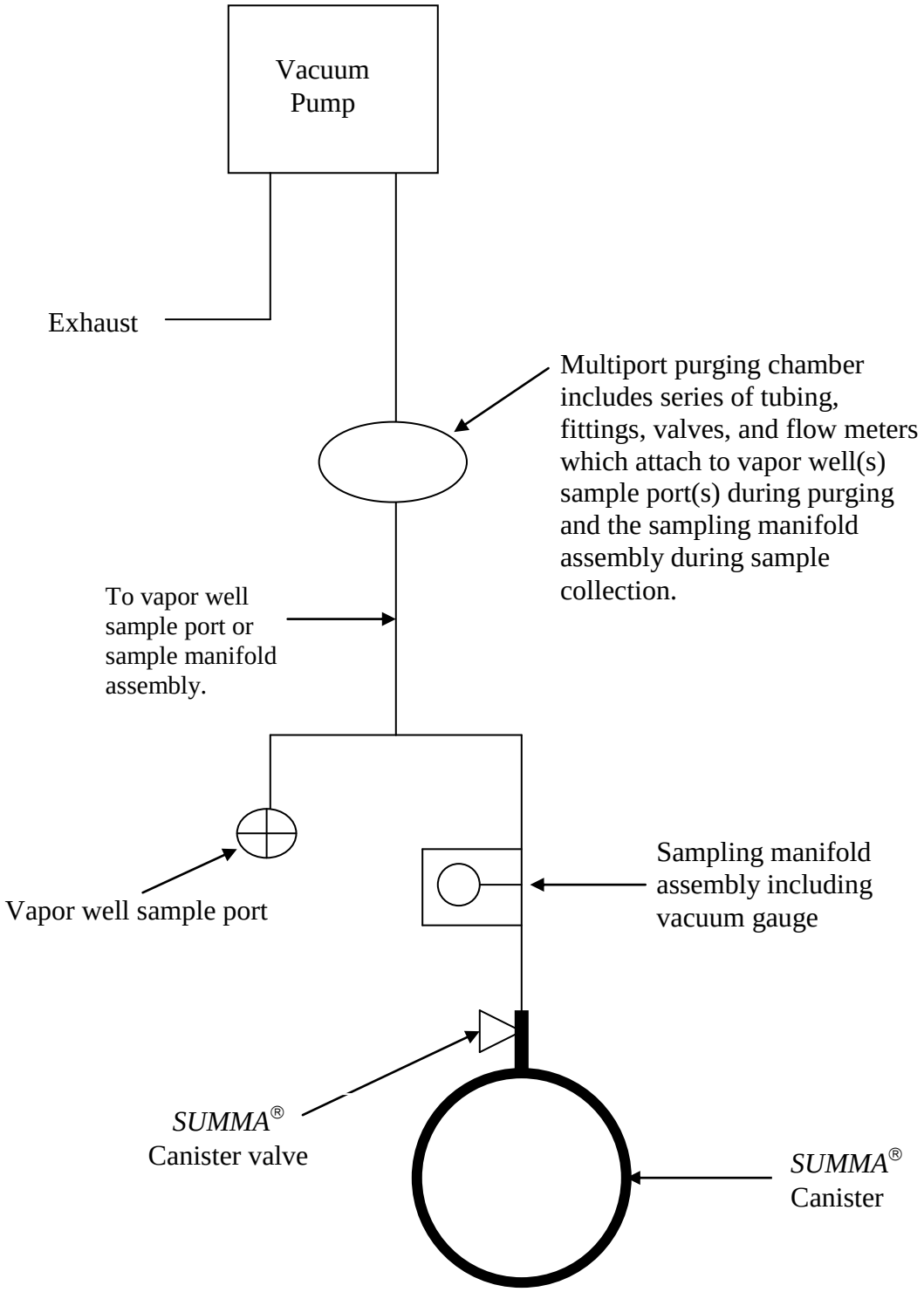
The TA-V soil vapor sampling equipment includes a sampling manifold assembly and a multiport purging chamber (Figure D-1). The multiport purging chamber is equipped with individual valves, fittings, and tubing which can be connected up to ten individual sample ports. The multiport purging chamber allows up to ten sampling locations to be purged at the same time. To setup the equipment and collect samples:

1) The ten valves on the multiport purging chamber are numbered 1 through 10. Connect the valve labeled #1 to the deepest sampling port. Connect the valve labeled #2 to the

second deepest sampling port. Continue connecting in this order with the valve labeled #10 connected to the shallowest sampling port.

2) Open valve on the multiport purging chamber associated with the shallowest sampling port and begin the purging process. Purging and sample from shallow to deep, as deep sampling ports may contain groundwater.
3) Turn on pump to purge soil vapor from sample depth.
4) Use the sampling tube volume and flow rate meter value to calculate the purge time for the sampling port. Purge for length of time that allows a minimum of three volumes of the sample tube to be evacuated.
5) Monitor the VOC levels of current sampling depth by attaching the VOC monitoring instrument of the exhaust port of the vacuum pump. Continue the purging process until the VOC levels stabilize. Record stabilized VOC reading on the Soil Vapor Sampling Log (FOP 08-22, Attachment F).
6) Close valve and turn off the vacuum pump.
7) Disconnect the multiport purging chamber valve from the sampling port.
8) Attach the sampling manifold assembly to the sampling port and to a SUMMA® canister.
9) Open SUMMA® canister valve.
10) Open flow valve on sampling manifold assembly by squeezing flow valve lever.
11) When the vacuum gauge on the sampling manifold assembly reaches approximately minus 10 in. Hg, release flow valve lever and close the SUMMA® canister valve. This will prevent the canister from going to ambient pressure (0 in. Hg). (Note: The analytical laboratory, Test America, requests that approximately minus 10 in. Hg of vacuum remains in the SUMMA® canister at completion of sampling).
12) Remove sampling manifold assembly from the SUMMA® canister.
13) Fill out date and time on sample label and attach it to SUMMA® canister tag. Do not attach sample label to canister itself.
14) Verify the final vacuum reading of approximately minus 10 in. Hg on all SUMMA® canisters by connecting the vacuum gauge provided by the laboratory to the valve on top of the SUMMA® canister.
15) Record the final vacuum reading for each canister on the SUMMA® Canister Log (FOP 08-22, Attachment E).
16) Close the SUMMA® canister valve, remove vacuum gauge and replace Swagelok® dust cap.
17) Proceed to the next sampling depth by opening the corresponding valve on the multiport purging chamber.
18) Continue with steps 4) through 16) until all sampling ports have been purged and samples have been collected.
19) If more than one sampling location or sampling port are purged at the same time, then modify purge length in step 4) as the length of time that allows a minimum of three volumes from each sampling tube to be purged based upon calculated purge time for the sampling port with the greatest volume.

Figure D-1
TA-V Vacuum Pump and SUMMA® Setup



TA-V Sampling Tube Volume and Purge Time Calculations

V of soil vapor screen = π * [(0.5 in.)^2 / 4] * 1 ft. * (12 in./ft.) * 1ft.^{3} / (12 in.)^3 = 0.0014 ft.^{3}
V of sampling tubing = π * [(0.25 in.)^2 / 4] * tubing length (ft.) * (12 in./ft.) * [1ft^3 / (12 in.)^3]
Vapor Well Volume = (V soil vapor screen + V of tubing)
V to purge = 3 * (Vapor Well Volume)
The sampling locations, associated ports, corresponding sampling depths, and purge volumes are presented in the table below.

Table D-1 TA-V Purge Volume Calculations

Sampling Locations	Sample Depth (bgs) tubing length (ft)	Soil Vapor Screen Volume (ft^3)	Sampling Tube Volume (ft^3)	Vapor Well Volume (ft^3)	Volume to purge (ft^3)
TAV-SV01 TAV-SV02 TAV-SV03	50	0.0014	0.0170	0.018	0.055
	100	0.0014	0.0341	0.035	0.106
	150	0.0014	0.0511	0.052	0.157
	200	0.0014	0.0682	0.070	0.209
	250	0.0014	0.0852	0.087	0.260
	300	0.0014	0.1023	0.104	0.311
	350	0.0014	0.1193	0.121	0.362
	400	0.0014	0.1364	0.138	0.413
	450	0.0014	0.1534	0.155	0.464
	500	0.0014	0.1704	0.172	0.515

Minimum pump running time to evacuate three volumes from each sampling port is calculated as follows:

t = V to purge / Q where: t = time
V = volume
Q = flow rate (to be determined in the field based on equipment limitations)

Attachment F

SUMMA® Canister Log

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SUMMA® Canister Log

Serial #	Date Received	Date Tested for Initial VAC	Initial VAC at 5400 ft (in. Hg)	Date Used	End VAC at 5400 ft (in. Hg)	Date Returned to SMO

SUMMA® Canister Log completed by:

Printed Name

Signature

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Attachment G

Soil Vapor Sampling Log

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Soil Vapor Sampling Log

Location	Date	Time	Canister #	PID (ppm)	Starting Vacuum (in. Hg)	Ending Vacuum (in. Hg)	Location Comments

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Attachment H

Soil Vapor Monitoring Inspection Form

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Soil Vapor Monitoring Inspection Form

1. Soil vapor monitoring site (CAMU, CWL, MWL, TA-V) _____

2. Date of Inspection _____

3. Time of Inspection _____

4. Name of Inspector _____

Mandatory requirement:

The inspector has read applicable site-specific Permits (CAMU and CWL), LTMMP (MWL) and activity related procedures in the last 12 months for the location indicated on line 1 above: (*Inspector must initial box before proceeding with the inspection.)*

Date read _____

Provide explanatory notes for each parameter not inspected or each action required. Include any remedial steps required.

SOIL VAPOR MONITORING LOCATIONS				
Inspection Parameter	***Indicate if Applicable (Yes or No)***	***Parameter Inspected (Yes or No)***	***Action Required (Yes or No)***	***Note Number***
A. Concrete pads, bollards, and protective casings in need of repair/maintenance.				
B. Above-ground enclosure in need of repair/maintenance.				
C. Well cover caps and Swagelok® dust caps in need of repair/maintenance.				
D. Sampling ports in need of repair/maintenance.				
E. Passive venting Baroballs™ in need of repair/maintenance.				
F. Monitoring wells and soil-gas sample port locations properly labeled.				
G. Locks in need of cleaning or replacement.				

Soil Vapor Monitoring Inspection Form (continued)

SAMPLING EQUIPMENT				
Inspection Parameter	***Indicate if Applicable (Yes or No)***	***Parameter Inspected (Yes or No)***	***Action Required (Yes or No)***	***Note Number***
A. Sampling pump in need of repair/maintenance				
B. Sampling manifold (tubing, gauges, and valves) in need of repair/maintenance.				

PREVIOUS DEFICIENCIES			
Inspection Parameter	***Parameter Inspected (Yes or No)***	***Action Required (Yes or No)***	***Note Number***
Uncorrected/undocumented previous deficiencies.			

NOTES

Note Number	Description

Soil Vapor Monitoring Inspection Form (concluded)

Action (Note Number) _____ assigned to _____Date action completed_____

Action (Note Number) _____ assigned to _____Date action completed_____

Action (Note Number) _____ assigned to _____Date action completed_____

Action (Note Number) _____ assigned to _____Date action completed_____

Action (Note Number) _____ assigned to _____Date action completed_____

Additional Comments:

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Inspector's Signature _____
Original to: Site's Operating Record
Copy to: Environmental Operations Records Center, MS-1309

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SANDIA NATIONAL LABORATORIES
LONG TERM STEWARDSHIP DEPARTMENT (4142)

GROUNDWATER MONITORING HEALTH AND SAFETY PLAN

PLA 05-09
Revision 05

Author: Tim Jackson, Subject Matter Expert — **Date:** 04/21/2014

Approved: Michael Skelly, Project Leader — **Date:** 21 APR 14

Approved: Don Schofield, Field Support Operations Project Leader — **Date:** 04/22/14

Approved: Pamela Puissant, Department Manager — **Date:** 06/16/14

Author: How frequently does this document need to be reviewed and/or revised?	Every **three** years, or when activities change.
Manager: Does this document need to be tracked?	**Yes**

EFFECTIVE DATE: _____

IMPORTANT NOTICE: A printed copy of this document may not be the document currently in effect. The official version is located on the Sandia Restricted Network (SRN), 4100 Controlled Documents home page.

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Revision History

Revision	Effective Date	Summary of Changes
1	10/10/2005	New document
2	8/16/2007	Formatting changes. Updated section 2, Roles and Responsibilities; section 3, Training Qualifications; section 11, References.
3	3/4/2010	Formatting changes. Added work planning and control information to section 6, Task Hazard Analysis. Updated section 3, Training Qualifications; section 11, References.
4	1/24/2012	Formatting changes. Revision history changed from 2 years to 3 years. Removal of some forms (attachments) and replaced with hyperlinks to where the forms can be found. Updated section 3, Training Qualifications; section 11, References.
5		Revised Work Planning and Controls requirements per Engineered Safety Implementation and included Mixed Waste Landfill Long Term Monitoring and Maintenance Plan HASP reference.

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ACRONYMS AND ABBREVIATIONS

AOP	administrative operating plan
EOC	Emergency Operations Center
ES&H	Environment, Safety and Health
FOP	field operating procedure
GMP	Groundwater Monitoring Program
HASP	health and safety plan
HNO_3	nitric acid
KAFB	Kirtland Air Force Base
LOP	laboratory operating procedure
LTS	Long Term Stewardship
OJT	on-the-job training
OSHA	Occupational Safety and Health Administration
PHS	primary hazard screening
PLA	plan
PPE	personal protective equipment
RCRA	Resource Conservation and Recovery Act
SAP	sampling and analysis plan
SNL/NM	Sandia National Laboratories, New Mexico
THA	task hazard analysis
TWD	technical work document

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1.0 PURPOSE, GOALS AND OBJECTIVES

Sandia Corporation conducts general groundwater surveillance monitoring for the U.S. Department of Energy (DOE), National Nuclear Security Administration (NNSA) at Sandia National Laboratories, New Mexico (SNL/NM). Monitoring is performed on a site-wide basis as part of the Long-Term Stewardship (LTS) Program's Groundwater Monitoring Program (GMP). The GMP includes groundwater surveillance and site-specific groundwater monitoring at LTS/Environmental Restoration (ER) Operations (formerly ER Project) sites with ongoing groundwater investigations.

This health and safety plan (HASP) covers groundwater monitoring operations that are detailed in associated regulations, requirements, and technical work documents (TWD) (i.e., administrative operating procedures [AOP], field operating procedures [FOPs], laboratory operating procedures [LOP], sampling analysis plans [SAP], and mini-SAPs).

Purpose The purpose is to recognize and anticipate all potential hazards associated with performing groundwater monitoring activities at SNL/NM.

Goals The goal is to perform groundwater sampling and surveillance activities with zero occupational injuries and reportable occurrences. The activities are described in detail in the associated TWDs.

Objectives The objectives are to perform work identified in the TWDs for groundwater monitoring activities by:

- Planning work so that potential hazards are recognized and controlled.
- Following health and safety protocols to prevent hazards to workers and protection of the environment.
- Executing work **only** as it is identified and described in the TWDs for groundwater monitoring activities listed in section 3.0. The work shall be performed in a manner that protects personnel from hazards, thus preventing injury.
- Limiting work activities to authorized and trained personnel.
- Improving this document and work processes (if necessary) based on feedback from personnel, safety case discussions, and lessons learned.

2.0 ROLES AND RESPONSIBILITIES

The Long-Term Stewardship Program is responsible for development, approval, distribution, revision, and control of this document.

The **Department Manager** is responsible for the following:

- Providing programmatic guidance leading to the development of this HASP.
- Reviewing and approving of this HASP.

The **Project Lead** or designee is responsible for the following:

- Reviewing and recommending approval of the HASP.
- Providing overall coordination and management of the GMP.
- Providing regulatory or programmatic requirements.
- Reviewing completed field forms and data pertaining to waste management and groundwater sampling activities.
- Reporting all information as required by regulations or directives.

The **Sampling Coordinator** is responsible for the following:

- Generating a mini-SAP from the SAP. The mini-SAP is a field friendly version of the SAP that:1) details sampling activities for the field support operations project leader and field technicians: 2) summarizes sampling procedures, analytical parameters, field measured parameters, purge requirements, and waste management tasks: and 3) identifies monitoring well characteristics that may extend the sampling period (e.g., low yield wells, well construction issues, etc.).
- Preparing a waste management plan for each sampling event.
- Providing the field support operations project leader with a copy of the mini-SAP.
- Coordinating waste management activities with the project leader and the field support operations project leader.
- Reviewing completed field forms and data pertaining to sampling activities.
- Ensuring that all data quality requirements are performed.
- Reviewing all analytical data used for waste characterization.
- Obtaining waste determination from the environment protection representative (non-regulated, hazardous, and radioactive).
- Managing, coordinating, and disposing of purge water and other waste generated from field operations.
- Obtaining discharge permits for purge and decontamination water from the Environmental Programs Water Quality Group.
- Submitting disposal requests to SNL Waste Management (Hazardous Waste Management Facility Radioactive and Mixed Waste Management Facility, and Solid Waste).
- Coordinating with the field support operations project leader for disposal and discharges.
- Tracking and documenting each waste activity.
- Performing and documenting weekly inspections of Building 9925 Resource Conservation and Recovery Act (RCRA) Less Than 90-Day Waste Accumulation Area.

- Performing and documenting weekly inventory of all waste stored at Building 9925 waste accumulation areas.
- Performing monthly inspection of emergency equipment.
- Maintaining documentation for waste disposal activities.
- Submitting completed field forms to the Customer Funded Record Center and entry of relevant data to the Environmental Data Management System (EDMS) database.
- Reviewing and providing recommendations for revisions to this plan (if necessary).

The **Field Support Operations Team Leader** is responsible for the following:

- Communicating with the sampling coordinator regarding sampling activities.
- Supervising the field technicians.
- Reviewing training requirements for field technicians.
- Providing on-the-job training (OJT) of new field technicians.
- Assigning field technicians (qualified by training and experience) to conduct the activities described in this HASP.
- Coordinating sampling activities with the sampling coordinator, Sample Management Office and field technicians.
- Providing field technicians with necessary equipment and supplies to conduct field work.
- Maintaining, reviewing, and revising all TWDs.
- Owner/manager/emergency coordinator of Building 9925 RCRA Less Than 90-Day Waste Accumulation Area.
- Reviewing and providing recommendations for revisions to this plan (if necessary).

The **Field Technicians** are responsible for:

- Stopping work if any operation threatens worker or public health and safety.
- Conducting tasks as described in the TWDs.
- Completing all necessary and required training as specified by the field support operations project leader.
- Conducting a tailgate safety meeting prior to the start of all field activities.
- Participate in the work Planning and Controls process, including the safety case discussions.
- Inspecting and maintaining equipment.
- Collecting, storing, and delivering samples to the Sample Management Office (SMO) in accordance with the SMO TWDs.
- Managing and disposing of waste as directed by completed Work Request Forms, (FOP 05-04, Attachment A) and the field support operations project leader.
- Performing project inspections in accordance with associated TWDs.
- Completing and reviewing field documentation forms.
- Providing recommendations for revisions to this plan (if necessary).

The **Training Director** is responsible for:

- Reviewing, verifying, and documenting the completion of all training required for groundwater monitoring activities.

3.0 TRAINING QUALIFICATIONS

Personnel conducting field activities shall complete the following:

- Read applicable sections of SNL/NM Corporate Policy ESH100 Environment Safety & Health
- Read primary hazard screening (PHS) SNL05A01241, *Long Term Stewardship (LTS) Groundwater Monitoring Activities*.
- Read and sign ALW 13-01, Activity Level Work Evaluation Form for the Groundwater Program.
- Read and sign AOP 95-16, *Sample Management and Custody*.
- Read and sign LOP 94-03, *Sample Handling, Packaging and Shipping*.
- Read and sign FOP 05-01, *Groundwater Monitoring Well Sampling and Field Analytical Measurements*.
- OJT for new field personnel performing groundwater monitoring activities if it pertains to any of the FOPs listed below. Document training by completing On-the-Job Training form (EP 2009-OJT).
- Read and sign FOP 03-02, *LTS Groundwater Level Data Acquisition and Management*. (Note: The training requirements denoted with an "*" in Table 1 below are all that are required for FOP 03-02.)
- Read and sign FOP 05-02, *Groundwater Monitoring Equipment Field Check*.
- Read and sign FOP 05-03, *Groundwater Sampling Equipment Decontamination*.
- Read and sign FOP 05-04, *Groundwater Waste Management Plan.*
- Read and sign FOP 09-05, *Conducting Slug Test Using Data Logger & Pressure Transducer* (only necessary if conducting slug test).
- Read and sign FOP 10-01, *Borehole and Downhole Well Video Inspection*. (Note: The training requirements denoted with an "*" in Table 1 below are all that are required for FOP 10-01).
- Read and sign plan (PLA) PLA 13-02, *Mixed Waste Landfill Long-Term Monitoring and Maintenance Health & Safety Plan*.
- Complete training courses listed in Table 1.
- Field personnel shall sign the Authorized Users List (EP 2009-AUL) to affirm they have read and understand this document, and agree to operate within the stated constraints.

Table 1. Training Course List

Course Code	Course Title
*CHM100/103	Chemical Safety Training/Site-Specific Chemical Training
*ELC105	Basic Electrical Safety (> 50 volts)
ELC901	Safe Switching Briefing
ENV100	OSHA Health & Safety Basic Training - General Worker (40 HR)
*ENV103	OSHA Health & Safety Training Refresher (8 HR)
*ENV112	Hazardous Waste & Environmental Management Training
ENV216	RCRA - Less Than 90-Day Area Accumulation Area for Owners & Emergency Coordinators
ENV316	RCRA - Less Than 90-Day Area Accumulation Area for Waste Workers
ENV416	RCRA - Less Than 90-Day Area Accumulation Area for Waste Workers - Site-Specific
*ESH100	ES&H Awareness
FKL153	Forklift Operator and Hands-On Training
*MCH200	Hand and Power Tool Safety
*MED102	Standard First Aid
*MED104	Heartsaver CPR
*OTS101	Occupational Thermal Stress
PKX100	Basic Hazardous Material Transportation Training
*PPE106	Personal Protective Equipment Training
PRS150	Pressure Safety Orientation
PRS250	Advanced Pressure Safety
*RAD102	General Employee Radiological Training
RAD230	Radiological Worker II Training

NOTES: *Training requirements are all that are required for FOP 10-01
CPR = Cardiopulmonary Resuscitation
ES&H = Environment, Safety and Health
HR = hour
OSHA = Occupational Safety and Health Administration
RCRA = Resource Conservation and Recovery Act

4.0 SCOPE OF WORK

The scope of work covered by this HASP **only** includes the activities as they are identified and described in the TWDs listed in Section 3.0. This HASP **can not** be utilized for any other work without the explicit authorization from the field support operations project leader or higher authority.

5.0 PERSONAL PROTECTIVE EQUIPMENT

Personnel are required to wear the personal protective equipment (PPE) identified by the task hazard analysis described in Section 6.0. Level D PPE will be the minimum level of protection for all activities (Table 2).

6.0 TASK HAZARD ANALYSIS

All activities associated with this HASP shall be covered by the PHS. Activity Level Work Evaluation Form, ALW 13-01, was completed and approved according to AOP 09-10, *Work Planning and Controls*.

Task hazard analyses (THA) have been performed on all groundwater activities in conjunction with PHS SNL05A01241, *LTS Groundwater Monitoring Activities*. The PHS helps identify potential hazards that can be expected when performing the work. The THA classifies the potential hazards and rates them based on the probability of occurrence (Table 2). The THA identifies control measures that will be used to mitigate the potential hazards (Table 2). The control measures may include courses and training that are identified as part of the PHS results. This approach to identifying, rating, and controlling hazards is consistent with SNL/NM's Integrated Safety Management System. The hazards rating are low for all activities identified in groundwater monitoring activities. Field technicians shall comply with PLA 13-02 during groundwater activities at the Mixed Waste Landfill, applicable FOPs when performing groundwater monitoring, in addition to requirements listed in this HASP.

Hazard assessment surveys were performed for groundwater sampling activities by an SNL/NM industrial hygienist. The following hazard assessment survey reports concluded that the potential for exposure to health hazards has been categorized as well-controlled; therefore acceptable:

- SNLNM00825, *9925/1 (High-Bay)* (equipment decontamination)
- SNLNM00827, *Groundwater Monitoring: Roving* (groundwater sampling)
- SNLNM01481, *Groundwater Surveillance: Roving* (measuring groundwater levels)
- SNLNM01520, *Chemwaste Landfill Groundwater Surveillance: Roving* (measuring groundwater levels at the Chemical Waste Landfill)

Groundwater monitoring consists of taking water samples from wells located on Kirtland Air Force Base (KAFB) and SNL. The following is the general order in which theses activities are performed:

- Equipment decontamination
- Calibration of monitoring equipment
- Collecting a depth-to-water measurement
- Lowering of pumps and or monitoring equipment
- Operating pumping equipment to purge the well (or sample line)
- Monitoring (measuring) chemical properties of water
- Operating pumping equipment to fill sample bottles
- Raising pumping equipment after samples have been collected
- Managing samples
- Managing waste water
- Documenting all activities

Table 2. Task Hazard Analysis - *Level of Protection* – Level D PPE (safety shoes/boots, chemical safety goggles)

Potential Hazard	Hazard Rating	Control
Chemical 1) Decontamination of pump tubing using a diluted nitric acid (HNO_3) rinse and a detergent rinse. 2) Groundwater containing volatile organic compounds (VOCs), nitrates & nitrites (20 parts per million (ppm). 3) Sample preservatives include sodium hydroxide (NaOH), hydrochloric acid (HCl), HNO_3, and sulfuric acid (H_2SO_4). Standardized solutions include Zobell solution, potential of hydrogen (pH) buffers, electrical conductivity solution. Other chemicals include various Hach ACCU-VAC ampules. 4) Spill of liquids (water) during operation of drum handler	Low	Wear chemical safety goggles and latex or nitrile gloves when handling potential chemical hazards. Portable eyewash is located in sampling vehicle. No eating, drinking, and smoking will be permitted around sampling operations. All purge water is treated as a non-regulated waste (based on process knowledge of prior sampling) until analytical results show otherwise. Material Safety Data Sheets (MSDSs) kept in sampling vehicle or obtained through the SNL/NM Chemical Information System. When discharging (dumping) drums requires mandatory 2-person team and follow guidance in the equipment manual provided by the manufacturer.
Mechanical 1) Motorized reel for raising and lowering pump. 2) Hydraulic lift on back of sampling vehicle.	Low	Be aware of potential pinch points. Do not wear loose fitting clothing, dangling badges or jewelry when operating this equipment.

Table 2. Task Hazard Analysis - *Level of Protection* – Level D PPE (safety shoes/boots, chemical safety goggles) (concluded)

Potential Hazard	Hazard Rating	Control
Mechanical (continued) 3) Operation of forklift. 4) Operation of drum handler	Low	Keep equipment within maintenance schedule and compliance. Keep current with training requirements. Requires use of "spotter" personnel when deemed necessary for safe operations.
Physical 1) Heat exhaustion & hypothermia. 2) Sunburn. 3) Lifting injury from equipment, pumps, and 55-gallon drums containing purge water. 4) Operation of water level meter (shoulder strain). 5) Lowering and raising pump (back strain). 6) Slips, trips, and falls. 7) Tools.	Low	1) Weather conditions are addressed in Tailgate Safety Meeting. Workers trained on heat exhaustion & hypothermia. Wear appropriate clothing and hydrate as necessary. 2) Provide workers with sunscreen. 3) Use proper lifting techniques. Utilize hydraulic lift on back of sampling vehicle and a forklift with a SNL/NM approved drum handler. 4) Use water level meter support device. 5) A motorized reel is used to lower and raise the pump. 6) Maintain proper housekeeping of work area. Use step stools. 7) Use correct tools and inspect them prior to use.
Radiological	Low	None are expected although a minimum of (Radiological Awareness or Radworker I) training is required.
Fire	Low	Fire extinguishers are located in mobile equipment.
Biological • Snakes, rodents, insects	Low	Care will be taken to observe that the well casings pose a potential for insects and other animals. Immediate area around wells will be kept clean and places of refuge for biological hazards minimized.

7.0 WORK PRACTICES

The following work practices will be enforced:

- All personnel must comply with OSHA, U.S. Department of Energy, and SNL/NM requirements regarding health and safety.
- No task will be performed until a PHS and THA has been prepared and reviewed with the personnel performing the task.
- All personnel must conduct their activities in a manner pursuant to the contents of this HASP.
- A tailgate meeting will be held prior to starting the day's sampling activities.
- Any unnecessary contact with potentially contaminated substances must be avoided. This includes contact with potentially contaminated surfaces and/or equipment.

- Eating, drinking, smoking, chewing gum or tobacco, or any other hand-to-mouth activities are prohibited in the sampling vehicle lab.
- A "buddy system" is implemented for all groundwater sampling activities. A "buddy system" is defined as a system of organizing personnel into work groups in such a manner that each member of the work group is designated to be observed by at least one other member in the group. The purpose of the "buddy system" is to provide rapid assistance to sampling personnel in the event of an emergency. In addition, a person is required to report his/her destination when leaving the other team member(s).
- All members of the sampling crew will carry a cell phone or portable radio capable of contacting the Emergency Operations Center (EOC).
- All members of the sampling crew will carry an EOC pager so they can be notified of any KAFB emergencies or weather alerts.
- An ABC fire extinguisher will be located in each of the field sampling vehicles.
- An eyewash device will be located in each of the sampling vehicles.
- A First Aid kit will be located in each of the sampling vehicles.

8.0 TAILGATE SAFETY MEETING

A field technician or field support operations project leader must conduct a tailgate safety meeting and fill out a Tailgate Safety Meeting Form (Attachment A) prior to the start of groundwater sampling activities. The person conducting the meeting must possess knowledge of groundwater sampling activities and the topics discussed in this HASP. All personnel/visitors who attend the meeting must document that they have attended and understood the meeting by signing the Tailgate Safety Meeting Form.

9.0 SHUTDOWN OF WORK ACTIVITIES

All individuals have the authority to shutdown groundwater monitoring activities if they feel that safety is being compromised. A shutdown could be the result of the following:

- Personnel not following health and safety protocols.
- Not having the appropriate safety gear on site (eyewash, first aid kit, fire extinguisher, appropriate PPE).
- Inadequate equipment or equipment failure.
- Weather
  - If lightning is observed within 5 miles (25 seconds from time of flash to thunder) or the EOC issues a lightning warning (via EOC pager).
  - High winds (greater than 40 miles per hour).
  - Severe snow storms (discretion of sampling crew).
  - Severe rain storms (discretion of sampling crew).
  - Severe heat or cold (discretion of sampling crew).
  - Tornado warnings.
- Unsafe conditions around sampling location (discretion of sampling crew).

- Any unsafe condition(s) noted during performance of groundwater monitoring activities or off-normal events.

In the event that work is stopped due to:

- Safety-related issues,
- an injury incurred while performing the tasks identified in this procedure, or
- as the result of an audit,

the field technicians shall immediately notify the sampling coordinator, field support operations project leader, the project leader, and the department manager. The field technicians shall seek the assistance of the field support operations project leader for the mitigation of the hazard and the completion of a Work Resumption Authorization Form (EP 2009-WRA) as required by AOP 09-10, *Work Planning and Control*. The department manager shall sign the completed form prior to the restart of work.

10.0 EMERGENCY RESPONSE PLAN

During groundwater monitoring activities, the potential for fire, explosion, or unplanned release of radionuclides or RCRA regulated hazardous waste or waste constituents that would significantly threaten human health or the environment is very low. In the unlikely event of an emergency, the SNL/NM EOC will provide coordination, resources, and appropriate emergency equipment on an as-needed basis. In case of an emergency:

- Stop work.
- Alert other personnel in the affected area.
- Evacuate the immediate area.
- Notify the appropriate resources or points of contact listed in Table 3.

Table 3. Points of Contact and Emergency Telephone Numbers

Resources and Contact	Telephone Number
SNL/NM Incident Command System (Fire, Ambulance, etc.)	911 (505-844-0911 from cell phone)
SNL/NM Medical Clinic	845-8159
SNL/NM Non-Emergency Number	311 or 844-6515
Poison Control Center	272-1222
Sandia Security / Key Service	North: 844-4657 South: 845-3114
ES&H concerns	844-6515
National Response Center (Environmental Emergencies)	800 822-9761
Personnel to Notify if an Incident Occurs	
SNL/NM Project Leader Michael Skelly	office: 845-7697 mobile: 270-5170
SNL/NM Department Manager Pamela Puissant	office: 844-3185 mobile: 239-9144
Center 4100 ES&H Coordinator Noel Duran	office: 284-9707 mobile: 270-8822

10.1 Directions to Medical Facilities

Directions to SNL/NM Medical Facility: From Technical Area I (**TA-I**) proceed to Harding Boulevard and/or Wyoming Boulevard. On Hardin Boulevard proceed west to Wyoming Boulevard. Turn right (north) on Wyoming Boulevard and travel north to F Street. Turn right (east) on F Street and proceed to 7th Street. The medical facility is located at the west end of Building 831 at the intersection of F and 7th Streets.

From Technical Area II (**TA-II**) proceed to East Ordnance Road. Proceed west on East Ordnance Road to Wyoming Boulevard. Turn right (north) to F Street. Turn right (east) on F Street and proceed to 7th Street. The medical facility is located at the west end of Building 831 at the intersection of F and 7th Streets.

From **Tijeras Arroyo** proceed to the Landfill Road. Proceed southwest on Landfill Road to Pennsylvania Street. Turn right on Pennsylvania Street and travel northwest to Wyoming Boulevard. Turn right (north) on Wyoming Boulevard and travel north to F Street. Turn right (east) on F Street and proceed to 7th Street. The medical facility is located at the west end of Building 831 at the intersection of F and 7th Streets.

See Attachment B for SNL Medical Clinic location map.

11.0 REFERENCES

Sandia National Laboratories, Corporate Policy ESH100 Environment Safety & Health, SNL/NM.

Sandia National Laboratories, Sample Management Office, AOP 95-16, *Sample Management and Custody* (latest edition), SNL/NM.

Sandia National Laboratories, Sample Management Office, LOP 94-03, *Sample Handling, Packaging and Shipping* (latest edition), SNL/NM.

Sandia National Laboratories, Environmental Programs and Assurance Department, FOP 03-02, *Long Term Environmental Stewardship Water Level Data Acquisition and Management* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, FOP 05-01, *Groundwater Monitoring Well Sampling and Field Analytical Measurements* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, FOP 05-02, *Groundwater Monitoring Equipment Field Check for Water Quality Measurements* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, FOP 05-03, *Groundwater Monitoring Equipment Decontamination* (latest edition), SNL/NM.

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Sandia National Laboratories, Long-Term Stewardship, FOP 09-05, *Conducting Slug Test Using Pressure Transducer and Data Logger* (latest edition), SNL/NM.

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Attachment A

Tailgate Safety Meeting Form

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TAILGATE SAFETY MEETING FORM

Dept: _____Well Location: _____Date: _____ Time: _____

Activities: _____
(Anyone has the right to cease field activities for safety concerns. The buddy system will be used when needed.)

Weather Conditions:
Temp: _____ °F Wind Speed: _____ MPH Humidity: _____ % Wind Chill ____ °F

Chemicals Used: Acids in sample containers, standard solutions, Hach ACCU-VAC ampules_____
Other: _____

Safety Topics Presented

☐ Be aware of slips, trips, and falls. Keep work area clean and use a stepping stool when necessary.		☐ Be aware of environmental conditions (heat / cold stress). Dress accordingly. Wear sunscreen if necessary. Stay hydrated.
☐ Wear safety boots.		☐ Be aware of electrical hazards
☐ Use safe lifting practices. Wear leather gloves if necessary.		☐ Be aware of pressure hazards.
☐ Be aware of pinch points on pump cable reel and hydraulic tailgate lift.		☐ No eating or drinking at sampling counter.
☐ Be aware of chemical hazards.		☐ Be aware of biohazards (snakes, spiders, etc.)
☐ Wear nitrile or latex gloves when sampling.		☐ Wear communication device (cell phone, EOC pager).
☐ Wear chemical safety goggles.		☐ Avoid spilling purge / decon water.

Hospital/Clinic: Sandia Medical Clinic Phone: 844-0911/911

Attendees

_____ _____
Printed Name Signature

_____ _____
Printed Name Signature

_____ _____
Printed Name Signature

_____ _____
Printed Name Signature

_____ _____
Printed Name Signature

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Attachment B

SNL Medical Facilities Location Map

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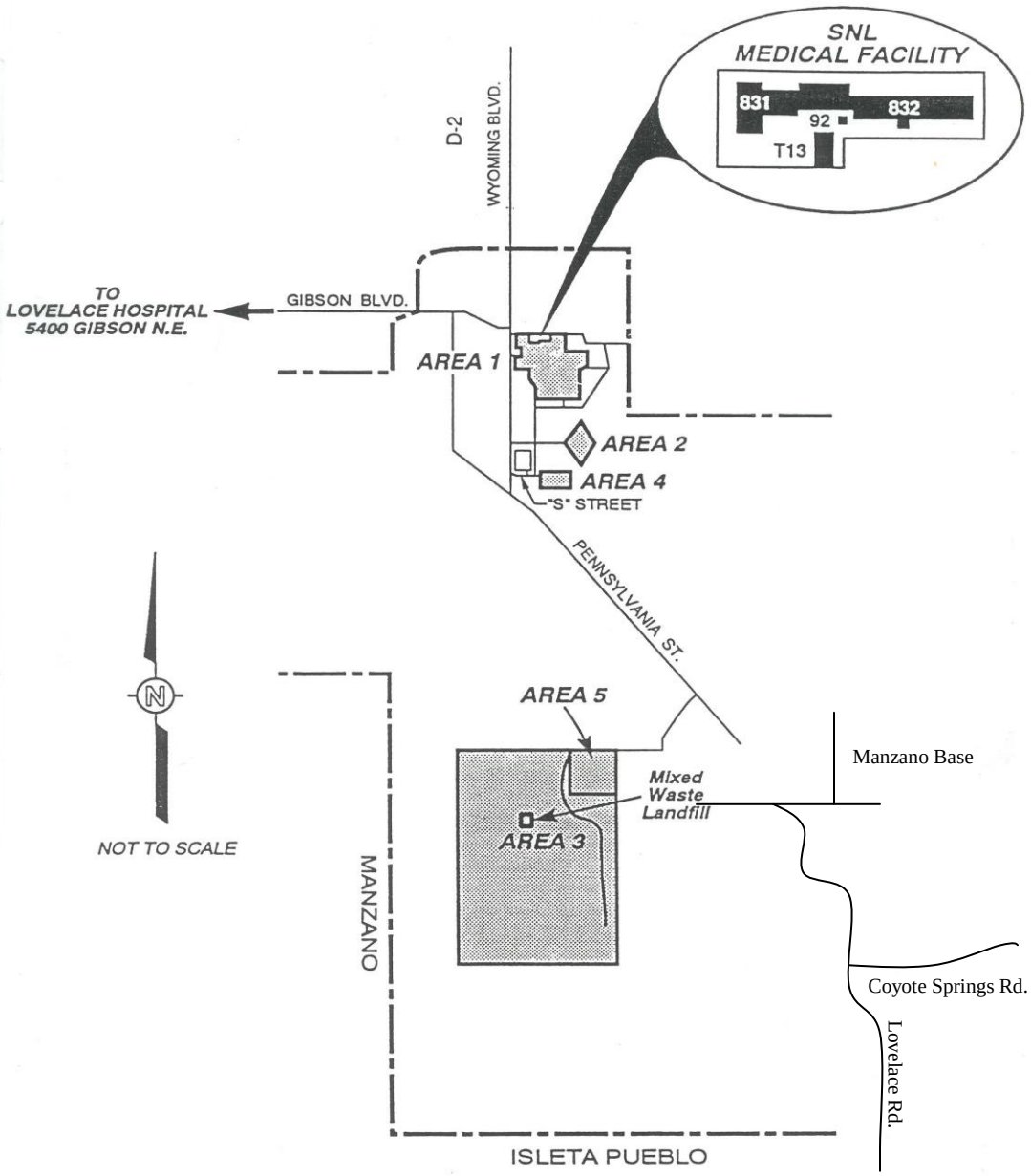

Figure 2
Medical Facilities Location Map, Sandia National Laboratories

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