National Nuclear Security Administration

Department of Energy
National Nuclear Security Administration
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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 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 reference documents to the New Mexico Environment Department (NMED) in accordance with Appendices C through G of the Mixed Waste Landfill Long-Term Monitoring and Maintenance Plan (LTMMP). This submittal is required within 60 days of NMED approval of the LTMMP, which occurred on January 8, 2014.

This submittal is comprised of procedures, plans and the analytical laboratory statement of work used by Sandia personnel for air, surface soil, soil vapor, soil moisture, biota, and groundwater sampling and analysis at the Mixed Waste Landfill (MWL) (total of thirteen documents). The cited documents are described in the following appendices to the MWL LTMMP:

Appendix C – Air Sampling and Analysis Plan for the Mixed Waste Landfill
Appendix D – Soil-Vapor Sampling and Analysis Plan for the Mixed Waste Landfill
Appendix E – Soil-Moisture Monitoring Plan for the Mixed Waste Landfill
Appendix F – Groundwater Sampling and Analysis Plan for the Mixed Waste Landfill
Appendix G – Tritium and Biota Sampling and Analysis Plan for the Mixed Waste Landfill

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

Sincerely,

James W. Todd
Assistant Manager for
Engineering

Enclosure
See Page 2

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

Sandia National Laboratories
Albuquerque, New Mexico
EPA ID No. NM5890110518

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

29 Jan 2014
Date signed

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

06 Mar 2014
Date signed

Enclosure A

Reference Documents Cited in the Mixed Waste Landfill Long-Term Monitoring and Maintenance Plan for Sandia National Laboratories/New Mexico (SNL/NM) Environmental Protection Agency Identification Number NM5890110518

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000

Index of MWL Long-Term Monitoring and Maintenance Plan Documents

Document Number	Title	LTMMP Reference
AOP 00-03	Data Validation Procedure for Chemical and Radiochemical Data	Appendix D (Table D-1-1), Appendix F (Table F-1-1), Appendix G (Table G -3.1-1)
AOP 95-16	Sample Management and Custody	Appendix C (Table C-4.1-1), Appendix D (Table D-1-1), Appendix F (Table F-1-1), Appendix G (Table G -3.1-1)
FOP 05-01	Groundwater Monitoring Well Sampling and Field Analytical Measurements	Appendix F (Table F-1-1)
FOP 05-02	Groundwater Monitoring Equipment Field Check For Water Quality Measurements	Appendix F (Table F-1-1)
FOP 05-03	Groundwater Monitoring Equipment Decontamination	Appendix F (Table F-1-1)
FOP 05-04	Groundwater Monitoring Waste Management	Appendix F (Table F-1-1)
LOP 94-03	Sample Handling, Packaging, and Shipping	Appendix C (Table C-4.1-1), Appendix D (Table D-1-1), Appendix F (Table F-1-1), Appendix G (Table G -3.1-1)
PLA 05-09	Groundwater Monitoring Health and Safety Plan	Appendix F (Table F-1-1)
SMO 05-03	Procedure for Completing the Contract Verification Review	Appendix D (Table D-1-1), Appendix F (Table F-1-1), Appendix G (Table G -3.1-1)
FOP 08-22	Soil Vapor Sampling	Appendix D (Table D-1-1)
NA	SNL/NM Statement of Work for Analytical Laboratories	Appendix D (Table D-1-1), Appendix F (Table F-1-1), Appendix G (Table G -3.1-1)
NA	Quality Assurance Project Plan for the Sample Management Office	Appendix D (Table D-1-1), Appendix F (Table F-1-1), Appendix G (Table G -3.1-1)
FOP 10-07	Field Operation Procedure for Soil Moisture Determination at the Mixed Waste Landfill Utilizing Neutron Logging	Appendix E (Table E-4-1)

Notes:

Appendix C = Air SAP for the Mixed Waste Landfill
Appendix D = Soil-Vapor SAP for the Mixed Waste Landfill
Appendix E = Soil-Moisture Monitoring Plan for the Mixed Waste Landfill
Appendix F = Groundwater SAP for the Mixed Waste Landfill
Appendix G = Tritium and Biota SAP for the Mixed Waste Landfill

Sandia National Laboratories/NM

Sample Management Office

DATA VALIDATION PROCEDURE FOR CHEMICAL AND RADIOCHEMICAL DATA

AOP 00-03

Revision 3

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List of Appendices

Revision History

Revision	Review Date	Effective Date	Summary of Changes
0	12/21/1999	12/21/1999	New document
1	12/08/2003	12/08/2003	Update to reflect current administrative changes
2	07/16/2007	07/16/2007	Update to reflect current administrative changes
3	05/12/2011	05/16/2011	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.

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 Environmental Protection Agency (EPA) 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 SMO Task Leader

The SMO Task Leader is responsible for:

- Ensuring this OP is implemented for review and validation of analytical data provided by the contract laboratories when data validation is requested.
- Providing oversight of the data review and validation process.
- Managing the validation contract, acting as the Sandia Delegated Representative

- Developing and maintaining processes that ensure the necessary documentation, to perform data review and validation, is made available to the data validator.
- Ensuring nonconformances are documented and corrective actions are initiated and closed.
- Managing a process for complete analytical data package archival to the Records Center

3.2 SMO Technical Data Support Staff

The SMO technical data support staff is responsible for:

- Completing the Contract Verification Review (CVR) form on analytical data packages from the contract analytical laboratories pursuant to "Procedure for Completing the Contract Verification Review," (SMO-05-03).
- Transmitting and tracking electronically the complete analytical data package (which includes the CVR form, analytical data, sampling documentation, supporting documentation, and if applicable laboratory Electronic Data Deliverable (EDD) to the data validation coordinator.
- Implementing and follow-up of all nonconformances and corrective actions with the contract laboratories and data validator.
- 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 QC checks on all data validation results by reviewing the report and comparing the results to the DV qualifiers captured on the EDD.
- Transmitting the complete analytical data package to the data administrator for final archiving.

3.3 SMO Data Administrator

The SMO data administrator is responsible for:

- Tracking analytical data delivered electronically from the contract analytical laboratories pursuant to "Procedure for Electronic Data Deliverable (EDD) Processing," (SMO-05-04).
- Forwarding the complete and final analytical data package and electronic data to the SNL/NM Records Center for archiving.

3.4 Data Validation Coordinator

The data validation coordinator is responsible for:

- Receiving, tracking, and distributing analytical data packages to data validators.
- Requesting data corrections or additional information needed from the analytical laboratories, notifying SMO of the request, and forwarding these to the data validator upon receipt from the laboratory.
- Notifying the SMO of nonconformances noted during the review process, and ensuring that nonconformances (e.g., incorrect or missing analytical information) are adequately addressed.
- Communicating with the SMO customer or designated representative when data review and validation is complete and sending the completed data validation report to the customer.

- Compiling the data validation report and checklists with the CVR form, analytical data package, sampling documentation, and if applicable validation EDD files (see Section 5.1 below), and returning the complete package to the SMO technical data support staff.

3.5 Data Validator

The data validator is responsible for:

- Reviewing the CVR form, analytical data, sampling documentation, and supporting documentation as described in this OP.
- Notifying the data validation coordinator and the SMO of all data determined as rejected (R coded) according to this OP.
- Notifying the data validation coordinator of any corrections or additional information required from the analytical laboratory.
- Completing the data validation report including checklist(s) and if applicable generating validation EDD files (see Section 5.1 below), and returning the data package and report to the data validation coordinator.

4.0 PROCEDURE

Data are evaluated using common quality parameters from quality control (QC) measurements specified in the methods and applicable contract statement of work (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 Contract Verification Review (CVR)

The SMO is responsible for conducting a CVR of analytical data packages delivered from the contract analytical 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 Analytical Request/Chain of Custody (AR/COC), receipt of samples by the laboratory, and the technical, QC, and reporting requirements imposed upon the analytical laboratory through the SMO contract SOW.

Evaluation	**Action**
The CVR form should be checked to confirm: AR/COC and laboratory login information have been reviewed, 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 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.	Report any discrepancies and/or anomalies associated with the CVR form to SMO.

4.1.2 Quality Control (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 (DO), 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 (MB/FB/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 C 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 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 initial calibration 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 method blank (MB), and 3) field blank (FB), equipment blank (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 DVR; for example, 125U, 18U, 9.9U, 0.32U or 0.032U.

4.1.12 Special Laboratory Flags

4.1.12.1 "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 SNL/NM SMO 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.

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 8000B). Contract Laboratory Program (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 laboratory control sample/ laboratory control sample duplicate (LCS/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 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 post-digestion spike; however, its analyte spike concentration may be higher than that of the MS/MSD. The post-digestion spike 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 detection limits and reporting limits 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

Data from independent reporting limit verification standards may be used to additionally evaluate the intercept. Acceptable reporting limit verifications may be used to minimize qualification based on professional judgment. An acceptable curve with a low standard at the reporting limit does not meet this requirement. The reporting limit verification 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 and 8270C.

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 initial calibration 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 (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 initial calibration 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 the Target 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,	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."
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,	qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ."
>3X the MDL,	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."
>2X the PQL,	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 initial calibration.

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 initial calibration 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 was 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 initial calibration shall be calculated according to the formula in Section 5.3 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 initial calibration 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.
>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

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

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 detection limit 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

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 8000B. 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 E 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.
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."
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 Internal Standard Performance

Internal standard (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 D 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 internal standard 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 8000B. 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."
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 laboratory control sample duplicate (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 8000B. 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	≤ 5 LCS fall outside acceptance criteria - no qualification
60 to 69 compounds	≤ 4 LCS fall outside acceptance criteria - no qualification
50 to 59 compounds	≤ 3 LCS fall outside acceptance criteria - no qualification
40 to 49 compounds	≤ 2 LCS fall outside acceptance criteria - no qualification
30 to 39 compounds	≤ 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 detection limits.
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.”
the sample was 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 were 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 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. 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 sample spectrum does not match the reference spectrum, the EICP RT or 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 Tentatively Identified Compounds (TICs)

Chromatographic peaks that are not target analytes, surrogates, or ISs are potential TICs.

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 (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,	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 relative retention time (RRT).
If sample TIC results are not sufficiently above the level in the blank and the results are reported,	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,	qualify that result as "NJ."
If a compound is not found in any blanks, but is a suspected artifact or common laboratory contaminant,	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, 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, 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 8280A, Polychlorinated Dioxins and Furans by GC/MS

Sample analysis shall be performed according to the requirements listed in SW-846 Method 8280A. 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 internal standard or target compound,	qualify all associated detects as "J" and all associated non-detects as "UJ."
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 internal standard 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 internal standard 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 internal standard solution compound is >150%,	qualify all associated detects as “J-” and all associated non-detects as “UJ.”
If the recovery of any internal standard 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%
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-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 8290, Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans by HRGC/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 initial calibration for unlabeled compounds and ≤30% of the mean values established during initial calibration 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 initial calibration. 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 initial calibration 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 is not met for any CCV compound at the beginning of a 12-hour shift, and…	
the %D is positive,	qualify all associated detects 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 as "UJ."
If the %D criteria were <u>not</u> met for any compound at the end of a 12-hour shift, a new initial calibration was analyzed within two hours of sample analysis, and…	
the %D is positive,	qualify all associated detects as "J+."
the %D is negative,	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 initial calibration 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%.

Evaluation	**Action**
If a replicate sample, MSD, 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 MS or LCS) and its replicate (or MSD 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."

Internal Standards

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 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-14, 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. In general, validate this analysis according to the Section 4.2. Also, this section provides some guidance that applies to TO-15.

Surrogates, an MS/MSD, and TICs are not required for this method.

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."
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 initial calibration 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 initial calibration shall be calculated according to the formula in Section 5.3 and must be $\leq$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 initial calibration RF and continuing calibration RF for any target compound is...	
>30% and positive (high bias),	qualify all associated detects as “J+.”
>30% but $\leq$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.

Internal Standard Performance

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

The retention time (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 D 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 internal standard area counts are <20% 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 >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 initial calibration, 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."
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,	qualify all associated detects as "J+."
the %R is below the verification 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 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 detection limits.
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 mm 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 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 initial calibration 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 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 initial calibration. 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.”

Internal Standards

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: A 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 detection limits.
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 initial calibration 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. **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 detection limits.
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 $\geq$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: 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 (internal standards). The RRF %RSD for the compounds calibrated using isotope dilution must be ≤20%. For the compounds calibrated using internal standards, 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 internal 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 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 internal standards must be within ±15 seconds of the RTs obtained during the initial calibration. 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 initial calibration 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 High-Performance Liquid Chromatography (HPLC) Validation

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

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 initial calibration 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 initial calibration 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."
<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,	qualify all associated detects <3X the absolute value of the intercept as "J-" and

>3X the MDL,	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."
>2X the PQL,	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 initial calibration. 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 initial calibration 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 initial calibration 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.
If the %D between ICV and/or CCV CF and the average CF obtained from the initial calibration 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

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

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 1: Data are not reported as 4U, as this would be reported as a detection limit 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

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 8000. 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. 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,	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 a surrogate %R is out of specification high,	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 8000B. 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 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,	qualify all associated detects as "J+."
either 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 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 3.3.5.

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 8000B. 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. f 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%.
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 detection limits.

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 initial calibration. 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	qualify the results for that compound in

sample requiring dilution exceeded the high calibration standard and was also found in the following sample at a concentration <5X the PQL,	the second sample as "R" or "NJ", based on professional judgment.
If <u>no data</u> 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.
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	qualify all non-detects from the diluted

the diluted sample data were reported,	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,	qualify the reported result as "J."
for high explosive (HE) analysis,	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 8082. When a primary column is specified, such as in SW-846 Method 8330, 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 8081A, 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 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 DDD and DDE as "NJ."
If DDT breakdown is >15% and DDT was	qualify the result for DDT in the sample

not detected in any sample analyzed after the last in-control standard but DDD and DDE were detected in any of those samples,	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 8082, PCB Aroclors by GC

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

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 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."
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 8015B, 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 internal standard is used to calculate analytical results, the slope or RF values are evaluated as directed below. If the analysis does not use an internal standard 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."
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 initial calibration standards must be ≤20%.

Evaluation	**Action**
For analyses using an internal standard 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 initial calibration 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 internal standard 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,	qualify all associated detects <3X the absolute value of the intercept as "J-" and all associated non-detects as "UJ."

>3X the MDL,	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."
>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 initial calibration. 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 initial calibration 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 initial calibration 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 initial calibration 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.
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 Reporting Limit Verification

A reporting limit verification standard (i.e., 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 of each HE analytical run as a measure of accuracy near the PQL. This analysis may be referred to as a post-digestion spike. 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+."
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

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

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 detection limit 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

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 internal standards 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 8000B. 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.
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 Internal Standard 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 internal standard area counts must not vary by more than 70% to 130% and the RT of the internal standard 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 internal standard is only used as an RT check, the RRT of the internal standard must fall within the acceptance range of 0.98 to 1.02, and the internal standard 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.
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%.

its RT varies by more than ±30 seconds,	**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. 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 internal standard 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 8000B. 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…	
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 8000B. 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."
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."
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.
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.

4.5 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 8330 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…	
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 Post Digestion Spike/CRI

If the original result is a detect, the LC/MS/MS result is a non-detect, and the LC/MS/MS post digestion spike/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 Internal Standard 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.”
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 initial calibration 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 (ICP)-atomic emission spectroscopy (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 (GFAA) for samples with analyte concentrations below 500 parts per billion (ppb).

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 (CVAA): 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 initial calibration,	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 SOW.

Criteria: The correlation coefficient (r) of the initial calibration 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, <0.90 but ≥0.80, <0.80,	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. 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."
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 any compound has an intercept that is positive and > the MDL,	qualify all associated detects <3X the intercept as "J+."

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 $\geq$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 affect 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.
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, post-digestion spikes (PS) 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	qualify all detects as "J" and all non-detects as "UJ."

above and below acceptance criteria,	
If the post-digestion spike %R is… >115%,	qualify all associated detects as "J+."
If the post-digestion spike %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."
Solid LCS If the LCS %R is… > the upper control limit, ≥30% but < the lower control limit, <30%,	 qualify all associated detects as "J+." 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 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 Reporting Limit Verification

Criteria: Reporting limit verification (i.e., CRA [AA], CRDL [cyanide], and CRI [ICP-AES, ICP-MS, and LC/MS/MS]) standards are analyzed at the beginning of each analytical run as a measure of accuracy near the reporting limit. 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 reporting limit verification 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 (ICS A and 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.
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+.”
<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

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

Criteria: A serial dilution must be analyzed for each matrix in an analytical run. If the undiluted results for the sample used for serial dilution 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 serial dilution 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."

Internal Standard (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 initial calibration 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 initial calibration 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, 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	Hardness	2.497 (2.5, if titrated)
Mg	Hardness (total hardness is the sum of the calculated Ca and Mg hardness results)	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 2.0mg/m^3 (2.5-1000ug/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 initial calibration is still valid.

Criteria: Calibrate the spectrometer according to the manufacturer's recommendations. Typically, an acid blank and a 10μg/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 initial calibration,	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+."
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 (µg/mL) is subtracted from the sample result (µg/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…	
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."

4.7 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-sigma).

Note: Some programs may request the result be reported with the 1-sigma uncertainty. When this is the case the reported uncertainty must be multiplied by two for evaluation of quantitation and 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-sigma 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 is > the MDA,	qualify the result as "R." **Note:** This action is not applied to gamma spectroscopy data.
If the absolute value of a negative sample result for gamma spectroscopy 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-sigma TPU and ≥ the MDA),	qualify all associated sample results ≥ the MDA but <5X the blank value as "NJ+."
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."
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-sigma 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-sigma control limits, and ±3-sigma 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-sigma limit, and ±3-sigma 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-sigma control limit when the laboratory provides sigma-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	qualify all associated results ≥ the MDA

lower control limit and a tracer is not measured simultaneously with the analyte (barium-133, neptunium-239, or thorium-234),	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) and a high-energy peak (> 1000 kilo electron volts). 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.
If the total result is ≤ the MDA and the partial result is > 2X MDA,	qualify the total result as "NJ-" due to a suspected false negative.
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 "J-."

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
- AR/COC 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
- AR/COC 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 AR/COC 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?
- Tentatively Identified Compounds (TICs). If required, are the identification and qualification of TICs discussed?
- Confirmation. Was second-column analysis discussed, and were qualifications properly applied?
- Reporting Limit Verification (RLV, CRI/CRA). Are all RLV and CRI/CRA 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 Serial Dilution (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?
- Laboratory Control Sample (LCS). Are all LCS issues that could affect data quality discussed, and qualifications properly applied?
- Matrix Spikes (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?
- Detection Limits/Dilutions. Is the appropriateness of the reported detection limits 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 (NCRs) that directly impacted data quality.
- Corrective Action Reports (CARs). 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 AR/COC numbers, SDG number, types and number of samples analyzed, and sample numbers.

5.5 CVR and AR/COC

The CVR and AR/COC 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 detection limit) - 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 detection limit 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 detection limit. 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 detection limit (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 detection limit (CRDL) or reporting limit (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 % Difference (%D) for LCSs, standards, and serial dilution 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 serial dilution

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 Replicate Error Ratio (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 sigma)
F_{95R} = replicate uncertainty (95% or 2 sigma)

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 initial calibration

7.0 ACRONYMS

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

AA	Atomic Absorption
AR	Analysis Request
BNA	Base/Neutral/Acid (Compounds)
BOD	Biological Oxygen Demand
CAR	Corrective Action Report
CAS	Chemical Abstract Service
CB	Chlorinated Biphenyl
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CF	Calibration Factor
CLP	Contract Laboratory Program
CRA	Reporting Limit Verification for AA Methods
CRDL	Contract-Required Detection Limit
COC	Chain of Custody
CRI	Reporting Limit Verification for ICP-AES and ICP-MS Methods
CVR	Contract Verification Review
CVAA	Cold Vapor Atomic Absorption
DL	Detection Limit
DO	Dissolved Oxygen
DOE	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
GFAA	Graphite Furnace Atomic Absorption
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
HT	Holding Time
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 Sample
ICS A	Interference Check Sample Solution A
ICS AB	Interference Check Sample Solution AB
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IS	Internal Standard
LAL	Lower Acceptance Limit
LC/MS/MS	Liquid Chromatography/Mass Spectrometry/Mass Spectrometry
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOC	Level of Chlorination
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
NCR	Nonconformance Report
ND	Not Detected/Non-detect

NIH	National Institute of Health
NIST	National Institute of Standards and Technology
OP	Operating Procedure
OPR	Ongoing Precision and Recovery
PAH	Polyaromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PCDD	Polychlorinated Dibenzodioxin
PCDF	Polychlorinated Dibenzofuran
PQL	Practical Quantitation Limit
PS	Post-digestion spike
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
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
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TPH	Total Petroleum Hydrocarbon
TPU	Total Propagated Uncertainty
UAL	Upper Acceptance Limit
VOC	Volatile Organic Compound

Measurements and Symbols

<	less than
≤	less than or equal to
>	greater than
≥	greater than or equal to
±	plus or minus
X	times
m	milli (1/1000)
%	percent
°C	degrees Centigrade
g	gram
ppb	parts per billion
ppbv	parts per billion by volume
k	Kilo
L	Liter
%D	percent difference
%R	percent recovery
%RSD	percent relative standard deviation
r	correlation coefficient
r^2	coefficient of determination
µ	micro (1/1000,000)
amu	atomic mass units

8.0 GLOSSARY OF TERMS

2-Sigma 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 1668 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: A low-level verification standard of the same origin as the calibration standard run as a measure of accuracy near the PQL. The reporting limit verification is known as the CRI for ICP-AES and ICP-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.

9.0 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 - Internal standard / tracer recovery failed high
IS2 - Internal standard / tracer recovery failed low but ≥10%
IS3 - Internal standard / 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-sigma TPU
FR4 - Negative result - absolute value >2X the MDA/MDL

FR5 - Retention time 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 - Reporting limit verification frequency not met[1]
DL2 - Reporting limit verification percent recovery failed high[1]
DL3 - Reporting limit verification percent recovery failed low[1]

CK1 - ICS frequency not met
CK2 - ICS analyte(s) failed high
CK3 - ICS analyte(s) failed low

D1 - Serial dilution 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.

10.0 REFERENCES

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Model Statement of Work for Analytical Laboratories (prepared for the DOE National Nuclear Security Administration Service Center)

U.S. Department of Energy NNSA Service Center Model Data Validation Procedure

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 HRGC/HRMS*

SW-846 Method 428, *Determination of Polychlorinated Dibenzo-p-dioxin (PCDD), Polychlorinated dibenzofuran (PCDF), and Polychlorinated Biphenyl Emissions from Stationary Sources*

SW-846 Method 8000B, *Determinative Chromatographic Separations*

SW-846 Method 8015B, *Nonhalogenated Organics Using GC/FID*

SW-846 Method 8081A, *Organochlorine Pesticides by Gas Chromatography*

SW-846 Method 8082, *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 8270C, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*

SW-846 Method 8280A, *The Analysis of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS)*

SW-846 Method 8290, *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 8330, *Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)*

SW-846 Method TO-14 (Rev. 1), *Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters with Subsequent Analysis by Gas Chromatography*

NIOSH 7300, Elements by ICP (Nitric/Perchloric Acid Ashing)

Appendix A

Data Reporting Requirements

NOTE: If any QC samples are analyzed using a different initial calibration 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 RPD acceptance criteria. The laboratory is not required to report calibration data associated with QC samples from another sample delivery group (SDG).

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
- Initial calibration data
- Applicable calibration verification data
- Continuing calibration check data
- Instrument and preparation blank data
- Surrogate data
- IS performance data
- MS/MSD data
- LCS data
- Sample results and analytical data for the requested target analytes, including results from dilutions, if analyzed
- Identification and data for any sample TICs
- Instrument run logs
- AR/COC and shipping documents
- Login worksheet
- Laboratory replicate data, if analyzed

Dioxins and Furans by HRGC/HRMS

- Case narrative
- Column performance check data
- Initial calibration data
- Applicable calibration verification data
- Continuing calibration check data
- Preparation blank data
- Labeled compound data
- 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
- AR/COC and shipping documents

- Login worksheet
- Laboratory replicate data, if analyzed

Gas Chromatography (GC) and High-Performance Liquid Chromatography (HPLC)

- Case narrative
- Initial 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
- AR/COC and shipping documents
- Login worksheet
- Laboratory replicate data, if analyzed

Polychlorinated Biphenyl (PCB) Congeners, EPA Method 1668A

- Case Narrative
- Initial calibration data, including 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)
- AR/COC and shipping documents
- Login worksheet

High Explosives (HE) and Perchlorate by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)

- Case narrative
- Initial calibration data

- Applicable calibration verification data
- ICB data
- Continuing calibration check data
- 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
- RT 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
- AR/COC and shipping documents
- Login worksheet
- Laboratory replicate data, if analyzed

Inorganic

- Case narrative
- Initial calibration 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
- ICP-AES and ICP-MS interference check sample (ICS) data
- ICP serial dilution data
- PQL verification (CRA/CRI/contract-required detection limit [CRDL])[2]
- Sample results and analytical data, including data from dilutions, if analyzed
- Instrument run logs
- AR/COC and shipping documents

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[1] CRI = reporting limit verification for LC/MS/MS, ICP-AES and ICP-MS methods.

[2] CRA = reporting limit verification for atomic absorption (AA) methods.
CRI = reporting limit verification for LC/MS/MS, ICP-AES, and ICP-MS methods.
CRDL = reporting limit verification for cyanide methods.

- Login worksheet

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
- AR/COC and shipping documents
- Login worksheet
- Control Charts

Appendix B

Sample Preservation and Holding Times

Sample Preservation Techniques and Holding Times

					Holding Times	
Method	**Parameters**	**Matrix**	**Volume/Container**	**Preservation**	**Sample**	**Extract**
305.1,310	Acidity, Alkalinity	Water	500 mL Plastic or Glass	4 °C	14 Days	NA
2320B	Bicarbonate					
300.0, 320.1,	Bromide, Chloride	Water	1 L Plastic	4 °C	28 Days	NA
325, 340, 375,	Fluoride, Sulfate					
9056						
405.1	BOD	Water	1 L Plastic	4 °C	48 Hours	NA
9010B, 9012A,	Total Cyanide	Water	1 L Plastic	4 °C; NaOH; pH > 12	14 Days	NA
9013, 9014,	Amenable Cyanide	Solid/Other	125 mL Glass Jar	4 °C	14 Days	NA
335.1, 335.3						
415, 9060	DOC, TOC	Water	250 mL Amber Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
200.7, 200.8,	All metals except Cr(VI) and Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	180 Days	NA
6010B, 6020,		Solid/Other	250 mL Glass Jar		180 Days	NA
7196A, 7197	Cr(VI)	Water	500 mL Plastic	4 °C	24 Hours	NA
218.6	Cr(VI)	Water	500 mL Plastic	4 °C, pH 9.3-9.7	28 days	NA
3060A,		Solid/Other	250 mL Glass Jar	4 °C	30 Days	7 days
245.1, 7470A	Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	28 Days	NA
7471A		Solid/Other	250 mL Glass Jar	4 °C	28 Days	NA
130.1, 2340B	Hardness	Water		HNO_3 ; pH < 2	180 Days	NA
				4 °C		
345.1	Iodide	Water	500 mL Plastic or Glass	4 °C	24 Hours	NA
353, 351,	Ammonium, Nitrate + Nitrite	Water	1 L Plastic	4 °C; H_2SO_4; pH < 2	28 Days	NA
365.4, 350	Total Phosphorus, TKN			4 °C; not acidified	24 hours	NA
300.0	Nitrate, Nitrite,	Water	500 mL Plastic	4 °C	48 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^{TM} 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
354.1	Ortho Phosphorus					
365	Ortho Phosphorus	Water	500 mL Plastic	4 °C; H_2SO_4; pH < 2	48 Hours	NA
9210/9211, 9056	Nitrate	Water	1 L Plastic	4 °C; 1M Boric Acid	48 Hours	NA
		Solid/Other	250 mL Glass Jar	4 °C	48 Hours	NA
314.0, 9058	Perchlorate	Water	250 mL Plastic or Glass	4 °C	28 Days	NA
6850 (modified)	Perchlorate by LC/MS/MS	Water/Solid	250 mL Plastic or Glass	4 °C	28 Days	NA
		Solid	4 oz. Wide-mouth jar	4 °C	28 Days	NA
410	Chemical Oxygen Demand (COD)	Water	250 mL Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
1664	Total Recoverable oil and Grease	Water	1 L Glass	4 °C; H_2SO_4 or HCl; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
9070/9071A	Total Recoverable Oil and Grease	Water	1 L Glass	4 °C; HCl; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
ASTM D-854	Specific Gravity					
376/9030B/9031	Sulfide	Water	1 L Glass	4 °C; NaOH; Zinc acetate; pH > 9	7 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	7 Days	NA
160	TDS, TSS, TS	Water	1 L Plastic	4 °C	7 Days	NA
160.4	Volatile solids (volatile residue)	Water	plastic or glass	4 °C	7 Day	NA
9020B	TOX	Water	1 L Amber Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
9060	TOC	Water	Glass	4 °C; H_2SO_4 or HCL; pH < 2 if analyzed >2 hours after collection	2 hours, unless acidified	N/A

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^{TM} 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
418.1	TPH	Water	1 L Amber Glass	4 °C; HCl; pH < 2	28 Days	NA
1664	TPH	Water	1 L Amber Glass	4 °C; H_2SO_4 or HCl; pH < 2	28 Days	NA
8440	TPH	Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
9065, 9066, 420	Total Recoverable Phenols	Water Solid	1 L Glass 125 mL Glass Jar	4 °C; H_2SO_4; pH < 4 4 °C	28 Days 28 Days	NA NA
150.1, 9040B	pH	Water	125 mL Plastic	4 °C	24 Hours	NA
110, 180.1	Color, Turbidity	Water	500 mL Plastic	4 °C	48 Hours	NA
120.1, 9050	Specific Conductance	Water	125 mL Plastic	4 °C	28 Days	NA
All radiochemical parameters except Rn-222 and tritium		Water Solid/Other	1 L Plastic (2 x 2 L Preferred) 250 mL Glass Jar	HNO_3; pH < 2	180 Days 180 Days	NA NA
913.0	Radon 222	Water	125 mL Glass	None	72 Hours	NA
906.0	Tritium	Water Solid/Other	1 L Glass Sample size will vary with moisture content		180 Days 180 Days	NA NA
8015 (Modified)	Petroleum Hydrocarbons (Diesel Range Organics)	Water Soil/Other	2 x 1 L Amber Glass Bottle 250 mL Glass Jar	4 °C 4 °C	7 Days 14 Days	40 Days 40 Days
	Petroleum Hydrocarbons (Gasoline Range Organics)	Water Soil/Other	3 x 40 mL Glass Vial 125 mL Glass Jar	4 °C; HCl; pH < 2 4 °C	14 Days 14 Days	NA NA
5035/8015 (Modified)	Petroleum Hydrocarbons (Gasoline Range Organics)	Soil	4 x 40 mL Glass Vial	4 °C, 2 Vials $NaHSO_4$ 1 Vial CH_3OH, 1 Vial No Preservative	*14 days	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^{TM} 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	 Extract
8021B	Halogenated Volatile Organics	Water	3 x 40 mL Glass Vial	4 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	4 °C	14 Days	NA
5035/8021B	Halogenated Volatile Organics	Soil	4 x 40 mL Glass Vial	4 °C, 2 Vials $NaHSO_4$ 1 Vial CH_3OH, 1 Vial No Preservative	*14 days	NA
8081A,	Organochlorine Pesticides	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
8082	PCBs	Water	4 L Amber Glass Bottle	4 °C	1 Year	1 Year
		Soil/Other	250 ml Glass Jar	4 °C	1 Year	1 Year
8141A	Organophosphorous Compounds	Water	4 L Amber Glass Bottle	4 °C; NaOH or H_2SO_4; pH 5-8	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
8151A	Chlorinated Herbicides	Water	4 L Amber Glass Bottle	4 °C;	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
8260B	Volatile Organics by GC-MS	Water	3 x 40 mL Glass Vial	4 °C; HCl; pH < 2	14 Days	NA
(modified)				4 °C; not acidified	7 days	NA
		Soil/Other	125 ml Glass Jar	4 °C	14 Days	NA 5035/8260B
	Volatile Organics by GC-MS	Soil	4 x 40 mL Glass Vial	4 °C, 2 Vials $NaHSO_4$ 1 Vial CH_3OH, 1 Vial No Preservative	*14 days	NA
8270C, 625	Semi-volatile Organics by GC-MS	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	14 Days	40 Days
8280A	Polychlorinated Dioxins and Furans by GC/MS	Water	4 L Amber Glass Bottle	4 °C	30 Days (1yr)	45 Days
		Soil/Other	250 mL Glass Jar	4 °C	30 Days (1yr)	45 Days
8318	N-Methylcarbamate Pesticides by HPLC	Water	4 L Amber Glass Bottle	4 °C; 0.1 $\underline{N}$ $ClCH_2CO_2H$, pH 4 - 5	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	7 Days	40 Days
8330	Nitroaromatics and Nitramines by	Water	4 L Amber Glass Bottle	4 °C	7 Days (1yr)	40 Days

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^{TM} 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
	HPLC	Soil/Other	250 mL Glass Jar	4 °C	14 Days (1yr)	40 Days
TO-13A	PAHs in Filter Cartridges	PUF, Tenax, or XAD-2 Filter Cartridge		4 °C	7 Days (1yr)	40 Days
TO-14A	VOC in Air	SUMMA® Canister			28 Days (by consensus)	
8321A (modified)	High Explosives by LC/MS/MS	Water	Amber Glass/Teflon lined cap	4 °C,	7 Days	40 Days
		Solid	Amber Glass/Teflon lined cap	4 °C,	14 Days	40 Days

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.

Appendix C

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 (cont.)
Maximum Holding Times
Non-detects in Water

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

Appendix D

GC/MS Internal Standards

Laboratories may vary the compounds calculated off of any 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 VOA 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 SVOA 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			

Appendix E

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
Semi-volatile 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
Semi-volatile 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
TCMX	81	25	138
Pesticides – Solid			
Decachlorobiphenyl	94	56	132
TCMX	97	69	124
PCB – Water			
Decachlorobiphenyl	88	42	133
PCB – Solid			
Decachlorobiphenyl	91	58	125
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 F

Laboratory Control Limits

Organic 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
2-Hexanone	591-78-6	92	56	128	97	47	146

Volatile Compound	**CAS #**	**Water**			**Solid**		
		Ave	**Low**	**High**	**Ave**	**Low**	**High**
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

Organic LCS Criteria Guidelines (Semi-Volatile Compounds)

Semi-Volatile 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
Diethyl phthalate	84-66-2	79	41	118	82	50	114

Semi-Volatile Compound	**CAS #**	**Water**			**Solid**		
		Ave	**Low**	**High**	**Ave**	**Low**	**High**
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

Organic LCS Criteria Guidelines (Pesticides and PCB)

Pesticide	CAS #	Water			Solid		
		Ave	**Low**	**High**	**Ave**	**Low**	**High**
Aldrin	309-00-2	83	27	138	93	47	140
α-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'-DDD	72-54-8	88	27	149	81	28	135
4,4'-DDE	72-55-9	87	33	140	97	68	126
4,4'-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

Organic LCS Criteria Guidelines

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

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.

RTs

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 (EICPs). 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.

RRTs

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 CCV).

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. GC/MS data systems using Target software automatically flag target analytes with a “Q” 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 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 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.

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Document Title: Data Validation Procedure for Chemical and Radiochemical Data

Document Number: AOP 00-03 **Revision:** 3

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SANDIA NATIONAL LABORATORIES
LONG TERM STEWARDSHIP DEPARTMENT (ORG 4142)

SAMPLE MANAGEMENT AND CUSTODY ADMINISTRATIVE OPERATING PROCEDURE

AOP 95-16
Revision 05

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Revision History

Revision	Effective Date	Summary of Changes
Rev 0	01/25/1995	New Document
Rev 1	04/08/1996	Administrative Updates
Rev 2	12/19/2003	Organization ownership change from Sandia ES&H to Environmental Restoration Project
Rev 3	03/28/2007	Changed revision cycle from 2 to 3 years. Organization ownership change from Sandia Environmental Restoration Project to Sandia ES&H Organization.
Rev 4	06/29/2011	Programmatic revisions include the addition of the Sample Management Analysis Request Tool (SMART) and the addition of Industrial Hygiene (IH) sampling. Other revisions are definition updates, sentence structure, grammar, and formatting. Additions include Revision History page, tracking box and footnote disclaimer
Rev 5	11/12/2013	Programmatic revisions include improvements to ARCOC processing, the addition of Bioassay sampling and changes to Industrial Hygiene (IH) sampling to include the use of the Radiological Process Knowledge Form (SF 6951-RRF). Chem101 and PKX050 were added to training requirements. The chemicals were removed from use in addition to removing the use of the fume hood. Added SMO QA Coordinator role. Other revisions include updating language to reflect current program elements and requirements.

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ACRONYMS AND ABBREVIATIONS

AOP	Administrative Operating Procedure
ARCOC	Analysis Request/Chain-of-Custody Record
COC	Chain-of-Custody
DOE	U.S. Department of Energy
DOT	U.S. Department of Transportation
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
IATA	International Air Transport Association
IH	Industrial Hygiene
LOP	laboratory operating procedure
OP	operating procedure
PHS	Primary Hazard Screening
RCT	Radiation Control Technician
RMA	Radioactive Materials Area
RPDP	Radiation Protection Dosimetry Program
RPPM	Radiation Protection Procedures Manual
RPSD	Radiation Protection Sample Diagnostics
SALI	Sample Analysis Laboratory Information
SARF	Sample Analysis Request Form
SMART	Sample Management Analytical Request Tool
SMO	Sample Management Office
SMO-QAPP	Sample Management Office Quality Assurance Project Plan
SNL/NM	Sandia National Laboratories/New Mexico
SOW	Statement of Work

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1.0 PURPOSE, SCOPE, AND OWNERSHIP

1.1 Purpose

This administrative operating procedure (AOP) describes the handling of samples at Sandia National Laboratories/New Mexico (SNL/NM) Sample Management Office (SMO) and delineates requirements for the selection of sample containers, required sample volumes, holding times, preservation techniques and sample custody control and documentation. This procedure also contains basic requirements for packaging and shipping environmental, industrial hygiene, bioassay and waste samples. Refer to the Sample Handling, Packaging and Shipping Laboratory Operating Procedure, LOP 94-03 for more detailed sample packaging and shipping requirements. This procedure implements Section 3.3.3, Sampling Handling and Custody Requirements, of the Sample Management Office/Quality Assurance Project Plan (SMO-QAPP).

1.2 Scope

This document applies to SNL/NM sampling projects that use the services of the SMO. Projects that reference this procedure or process samples through the SMO shall comply with this procedure. Samples, forms, and data submitted to the SMO for processing shall conform to the requirements in this procedure.

1.3 Ownership

The SMO owns this document. The SMO is responsible for preparing, revising, and distributing this document as necessary.

2.0 RESPONSIBLE INDIVIDUALS AND ORGANIZATIONS

The **Department Manager** is responsible for the following:

- Providing programmatic guidance leading to the development of this AOP.
- Reviewing and approving the procedure.
- Acting as liaison to the U.S. Department of Energy (DOE) and National Nuclear Security Administration/Sandia Field Office (NNSA/SFO) regarding sample management issues.

The SMO **Technical Lead** is responsible for the following:

- Updating this procedure.
- Developing and maintaining the SNL/NM Sample Management Office (SMO) Contract Statement of Work for Analytical Laboratories (SMO-SOW).
- Managing contractor laboratory services including procurement, routine performance assessments and general laboratory oversight.

The SMO **QA Coordinator** is responsible for the following:

- 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.

The **SMO Packaging Coordinator and Packaging support staff** are responsible for receiving and packaging samples shipped through the Receiving/Mail & Material Movement Organization (10263, "Shipping and Receiving") to the contracted laboratories for analysis. SMO Packaging Coordinator(s) and Support Staff responsibilities include but are not limited to the following:

- Overseeing the day-to-day operations of the SMO Sample Packaging Facility and support personnel.
- Verifying proper sample collection documentation from field sampling personnel.
- Ensuring that sample custody is properly maintained and documented in accordance with the most current SMO-QAPP.
- Ensuring all samples, with the exception of groundwater samples or known non-radiological samples with a Clearance-Radiological Process Knowledge Form SF 6951-RRF (Attachment A) on file, receive a radiological survey by a Health Physics Radiation Control Technician (RCT) prior to shipment to an analytical laboratory.
- Ensuring samples are properly stored and packaged for shipment to the analytical laboratories in accordance with the SMO-QAPP, DOE, U.S. Department of Transportation (DOT) and International Air Transportation Administration (IATA) regulations. (Refer to LOP 94-03.)
- Interfacing with SNL/NM Shipping, Radiation Protection Operations and other SNL/NM on-site organizations;
- Ensuring Samples are shipped in a timely manner giving laboratories sufficient time to analyze samples within holding times.

The **SMO Customer(s)/Sampling Personnel** are responsible for sampling and initiating chain-of-custody documentation. Sampling personnel are responsible for performing the applicable activities prescribed in this procedure, including but not limited to:

- Utilizing the Sample Management Analysis Request Tool (SMART) to initiate and submit bottle orders, to produce container labels and to produce and submit the Analytical Request Chain-of-custody (ARCOC) (Attachment B).
- Utilizing the Industrial Hygiene (IH) Sampling Analysis Request Form (SARF) chain-of-custody for industrial hygiene customers (Attachment C).
- Utilizing the Radiation Protection Dosimetry Program (RPDP) SARF chain-of-custody for RPDP customers (Attachment D).
- Providing the SMO with the Radiation Survey Documentation (Attachment H) for samples coming out of a Radioactive Material Area (RMA).
- Working with the Radiation Protection program, and providing the SMO with a copy of the Clearance-Radiological Process Knowledge Form, SF 6951-RRF (Attachment A), for non-

radiological samples. This is not mandatory, but will help expedite sample processing for some sampling programs.

- Labeling and using the correct containers and preservatives for the materials to be analyzed.
- Documenting field parameters during the sampling event according to applicable sampling procedure(s).
- Collecting sufficient volumes of samples for all analyses, including quality control analyses.
- Delivering samples to the SMO Packaging Facility according to chain-of-custody requirements and in secure/safe condition according to SMO requirements as stated in the Sample Handling, Packaging and Shipping Laboratory Operating Procedure (LOP 94-03).
- Delivering samples to SMO in a timely manner and communicating with SMO staff about short holding times.

The **Analytical Laboratory** is responsible for following the applicable SMO-SOW. Requirements include, but are not limited to:

- Developing and maintaining quality assurance programs and procedures in accordance with the applicable SMO-SOW.
- Providing sampling kits that include coolers and certified sample containers to the SMO using appropriate cleaning methods and preservatives.
- Ensuring that sample custody is properly maintained and documented in accordance with the current SMO-QAPP and SMO-SOW.
- Immediately notifying the SMO of non-conformances such as broken sample custody seals, leaking sample containers, broken sample containers, incorrect sample containers and incorrect preservation (i.e., pH, temperature).
- Performing analyses in accordance with the applicable SMO-SOW.
- Analyzing samples within required holding times.
- Providing data in accordance with format requirements in the applicable SMO-SOW.
- Handling of samples from receipt at the laboratory through completion of analysis maintaining sample integrity.
- Returning sample control documentation and analytical reports to SMO in accordance with contract and project requirements.

3.0 TRAINING QUALIFICATIONS

Personnel shall be trained and qualified as necessary to perform their assigned work. The Sandia Education and Training Organization provides basic training and qualification guidance. Training requirements are presented in activity-specific operating procedures with specific requirements for the tasks performed. Personnel shall be trained according to established training cycles to maintain proficiency. Training shall be updated to meet required frequency schedules when specified. Details of corporate training are outlined in the SMO-QAPP and are referenced in the current Primary Hazard Screening (PHS) document, PHS 972834764, SMO Packaging Facility Operations and in LOP 94-03.

SMO personnel and customers are responsible for adherence to training requirements stipulated in this procedure and the current SMO-QAPP and the SMO Packaging Facility Operations PHS as it pertains to each individual.

4.0 SAMPLE DOCUMENTATION AND MATERIALS

4.1 Bottle Order/Sample Request Form & Sample Containers

The Bottle Order/ Sample Request form is accessed through the SMART application. The completed and approved Bottle Order initiates the sampling process and is used to complete the ARCOC. The ARCOC may be obtained from the SMO home page, Electronic Forms link, or through the SMART application. Note that the SMART application is *not* used for the IH SARF or RPDP SARF chain-of-custody.

Recommended sample containers and chemical preservatives may be obtained by logging on to the SMO SMART application and completing the Bottle Order/Sample Request form. Most projects require that a bottle order be placed with the contract laboratory. (The IH and Bioassay projects do not utilize the SMO Bottle Order process.) It is recommended that bottle orders be submitted for approval to the SMO one to two weeks prior to sampling. The purpose of the bottle order is to:

- Initiate the sampling process.
- Notify the lab of the expected number of samples and analyses.
- Notify the lab of expected sampling dates.
- Obtain sample containers from certified suppliers with analytical method specified chemical preservatives. (Containers are inspected as covered in Part B, Section 3.3.8 of the SNL/NM SMO-QAPP.)

4.2 Analytical Request Chain-of-custody (ARCOC or COC, SARF) Forms

The COC provides an accurate and defensible written and/or computerized record to trace the possession and handling of a sample from collection to completion of all required analyses. COC records provide a record of sample history and are critical for data integrity. Four different COC forms may be submitted to the SMO:

- Contract Laboratory Analysis Request and Chain-of-custody (ARCOC) (Attachment B)
- Industrial Hygiene SARF (IH SARF)(Attachment C)
- Radiation Protection Dosimetry Program Sample Analysis Request Form (RPDP SARF) (Attachment D)
- Onsite Laboratory/Radiation Protection Sample Diagnostics (RPSD) Sample Analysis Request Form (RPSD SARF) (Attachment E)

4.3 Sample Label

An SMO SNL/NM sample label must be completed with indelible ink and affixed to each sample container prior to or during sampling. The label in Attachment F is required for all samples submitted on an ARCOC. The Sample Label is produced after completion of the SMART application ARCOC and using the label printer model Zebra LP 2844. It may also be produced using a spreadsheet and the Zebra label printer. A label printer is available for customer use at the SMO Packaging Facility (Building 928) and at the Field Office (Building 9925). The Sample Label information shall match the information on the corresponding ARCOC. For samples not submitted under the ARCOC, the label information must match the information on the SARF. Each completed sample label submitted on an ARCOC includes the following:

- SMO SNL/NM Sample Identification Number (The first 5 digits of the Sample Number are controlled and obtained from the SMO. The Sample fraction designation is assigned by the sampler.)
- ARCOC Number
- Sample location
- Date and time of sample collection
- Sample matrix type
- Chemical Preservative
- Analysis
- Collector's name
- SNL/NM Thunderbird logo

Samples submitted on a SARF chain-of-custody will contain a SARF chain-of-custody number. and Sample Number.

4.4 Custody Seals

Sample custody seals are used to help determine unauthorized tampering of samples following collection until the time of sample preparation and analysis. SNL/NM uses adhesive backed seals with the SNL/NM Thunderbird logo. Custody seals may be obtained from an SMO Packaging Facility representative. Initialed and dated seals must be affixed to sample containers before the samples leave the custody of the sampling personnel. IH containers and Volatile Organic Compound containers are exempt from this requirement. The container(s) are placed in a sealed plastic bag and the bag, not the container(s), is secured with the custody tape.

- The custody seal is initialed and dated while the seal is affixed to the backing.
- The seal is then removed from the backing and affixed to the container in such a manner that it is necessary to break the seal in order to open the container.
- The custody seal may be removed by the person initiating or retaining custody of the sample (e.g., the sampling personnel or the analytical laboratory sample custodian).
- The integrity of the seal must be verified prior to its removal.
- A broken seal invalidates the sample and must be documented as a nonconformance.

4.5 SNL/NM "Shipper" Form and Shipper's Waybill

The SNL/NM electronic shipping form, Web Shipper, is required on all shipments leaving SNL/NM. The Web Shipper and the commercial shipper's waybill complete the sample custody documentation and show possession of the sample from shipment to arrival at a contract laboratory. A copy of the Web Shipper form and shipper's waybill (if applicable) under which the samples are shipped shall be retained to document shipment of the sample(s). The SMO Packaging Facility Personnel are responsible for completing all shipping documentation per the current version of LOP 94-03.

5.0 SAMPLE MANAGEMENT PROCEDURES

5.1 Sampling Kit Procedure (Bottle Orders)

Required sample containers and chemical preservatives are obtained by logging on to the SMO SMART application and completing the Bottle Order/Sample Request form. Most projects require that a bottle order be placed with the contract laboratory. IH and RPDP Bioassay programs do not require a Bottle Order.

The Bottle Order initiates the sampling process for the projects that utilize the ARCOC. The customer shall submit a Bottle Order request to the SMO utilizing the SMART application. Upon receipt, the SMO reviews the Bottle Order and submits it to the contract laboratory. The contract laboratory provides sampling kits according to the Bottle Order specifications. The SMO shall ensure staff is trained in sampling kit requirements. It is recommended that bottle orders be submitted for approval to the SMO one to two weeks prior to sampling. IH and RPDP projects do not utilize the SMART application for sampling kits.

The SMO shall provide oversight and ensure that the laboratories follow the SMO-SOW as it pertains to providing sampling kits. The laboratories shall have applicable procedures and processes in place. The SMO shall inspect sampling kits to determine that they are intact, accurate, and meet any specific written requirements associated with the SMO-SOW. Any errors or damage to sampling kits will be addressed in accordance with the SMO-SOW and procurement policies.

5.2 Sampling Considerations

For sampling requirements, refer to Sample Containers and Preservatives, Holding Times and Sample Preservation (Attachment G).

Sample Volume**:** The volume of the sample collected should be sufficient to perform all the required analyses plus any additional volume needed to meet quality control requirements or repeat analyses. The minimum sample volume required for typical analytical procedures is listed in Attachment G, Holding Times and Sample Preservation. After the Bottle Order/Sample Request form is complete and approved, the required sample volumes will auto fill on the ARCOC when initiated. Laboratory-specific sample volume requirements may apply.

For the IH SARF chain-of-custody, the IH customer is responsible for meeting volume requirements.

Sample Preservation: Prior to sampling, the appropriate chemical preservative(s) is added to the sample bottles by the analytical laboratory. Due to the variety of chemical tests performed on samples, it may be impractical to chemically preserve samples during actual field collection. Following collection, most samples are cooled and maintained at <6˚C (i.e., stored in a cooler with ice or ice gel or in a refrigerator) to conform to temperature preservation requirements. Chemical and temperature Preservation requirements are listed in Attachment G and will auto fill on the Bottle Order and the ARCOC forms.

For the IH SARF COC, the IH customer is responsible for meeting sample preservation requirements.

Holding Times: Holding time is the time interval between sample collection and sample preparation or analysis. Holding times are calculated in days or hours, according to the time units used in the U.S. Environmental Protection Agency (EPA) holding time requirements. That is, if the EPA-specified holding time is given in hours, then the analysis must be complete before the end of the last hour of the holding time when calculated from the sampling time. When the holding time is given in days, the analysis must be complete before the end of the day on which the holding time would expire as calculated from the sampling day. Recommended maximum holding times are listed in Attachment G and should be adhered to. Samples should be shipped to the laboratory at the completion of each day of sampling, or as soon as practical.

SMO will make every effort to notify the laboratory when samples having less than 72 hours of the holding time remaining are to be shipped.

Sample Storage: All samples shall be stored in a secured location when not in the immediate custody of an individual. The samples should be stored under physical and environmental conditions commensurate with the preservation requirements and intended analysis. Sample integrity must be maintained during sample storage through access controls and documentation. Samples shall be placed in a sample storage refrigerator and allowed to equilibrate to the required temperature prior to shipment to a contract laboratory.

Daily verification and documentation of storage temperature should be maintained when temperature is a preservation requirement. Additional measures must be taken to separate waste samples from non-waste samples in order to avoid cross-contamination. Trip blanks should be used as appropriate to determine sample contamination during sample storage and shipment.

5.3 Sample Custody Procedure

Custody procedures provide an accurate record of sample history and shall be followed by SMO, field (sampling) and laboratory personnel to provide an accurate record of sample history.

By definition, a sample is in custody if it is:
- In one’s possession,
- In view,
- In a controlled access area
- In transit following proper chain-of-custody procedures.

Sampling Team Member (Customer) Custody Procedure
A Sample Team Member is responsible for the care and custody of samples collected until sample custody is properly transferred. The following procedure shall be used to ensure proper control of samples:
- For the ARCOC, a project team member must submit to the SMO a Bottle Order at least 7 days prior to the requested delivery date for containers. The Bottle Order is initiated using the SMART application found on the SMO homepage. Once the Bottle Order is submitted, it will go through an SMO approval process and will be submitted to the appropriate analytical laboratory. Upon approval, all project team members will be notified. The approved Bottle Order is used by the customer to

complete the ARCOC. From the approved Bottle Order, the Sample Matrix, Container Type and Volume, Preservative, Analysis Parameter and Method will pre-fill the ARCOC. SMART application Bottle Orders are not submitted for the IH or RPDP Bioassay SARF COC or the Onsite Laboratory (RPSD) Sample Analysis Request Form (RPSD/SARF).

- For the ARCOC, the Sample Team Member documents sample collection information: *Sample-No.-Fraction, Sample ID or Sample Location Detail, Pump Depth, Date/Time Collected, Collection Method, Sample Type, Filtered/Unfiltered sample* (refer to LOP 94-03).
- For the IH SARF COC, the sample team member will provide a completely filled form to include *IH Survey ID number, Submitted by name, Submission date, Analysis Requested, IH Sample #, Col. Date, Turn-Around-Time, Matrix.* In the *Sample Comments* section, the IH sample team member or the SMO will assign a unique sample number and fraction for each *IH Sample #.* The IH SARF COC is 2 pages. The second page is the received/relinquished page. The corresponding second page must include the IH Survey ID number.
- If samples are destined for the on-sight Radiation Protection Sample Diagnostics (RPSD) laboratory for analysis (i.e., gamma spec, alpha, beta, etc.), the customer shall complete either the paper form RPSD/SARF COC (Attachment E) provided by the RPSD laboratory or submit an electronic version of the SARF through the Sample Analysis Laboratory Information (SALI) system.
- For the ARCOC, sample container labels (Attachment F) shall be affixed to sample containers, or in some cases, to the re-sealable bag, and shall match information on the associated ARCOC. Sample labels shall be legible and completed in indelible ink. (Refer to LOP 94-03). Blank Sample Labels may be obtained by contacting an SMO Packaging Facility representative or may be printed from the SMO SMART Application. (See Sample Labeling section.)
- All samples shall be accompanied with corresponding ARCOC, IH or RPDP SARF, COC, and/or RPSD SARF documentation.
- Samples shall be delivered to the SMO Packaging Facility for review of custody documentation prior to acceptance and transfer of custody to the SMO. (Refer to LOP 94-03).
- The SMO Packaging Facility shall process samples as required by LOP 94-03. Samples submitted by sampling team members to the SMO Packaging coordinator or support staff shall be clean, sealed, and intact. Sample container lids shall be secured with custody tape that has been initialed and dated. Glass containers are placed in re-sealable bubble bags and double bagged. If samples are from an area designated as a Radioactive Material Area (RMA), the sampling team member shall include the Radiation Survey Documentation (Attachment H) with the samples. (Refer to LOP 94-03).
- A sampling team member shall assist the Sample Packaging Facility representative verifying that all sample containers and request forms are correct and complete. (Refer to LOP 94-03).
- Upon complete verification, the sampling team member shall transfer custody of the samples to the SMO Packaging Facility representative by signing, dating and noting the time on the appropriate *Relinquished By* line on the ARCOC,IH or RPDP SARF, COC, and/or RPSD SARF. The SMO Packaging Facility representative shall then accept custody by signing, dating, and noting the time on the appropriate *Received By* line, below the *Relinquished By* signature.

Packaging Coordinator Procedure

- The SMO Packaging Facility representative shall relinquish samples to the contract laboratory by signing the *Relinquished By* line on the ARCOC, SARF, or COC. The chain-of-custody documentation is then put into a zip-lock bag and placed inside the shipping container/cooler. The shipping container/cooler is then closed and sealed with custody tape and delivered to SNL/NM Shipping and Receiving personnel for shipment to contract laboratories. Included with the shipping container is a completed Web Shipper. The SNL/NM Shipping and Receiving Department shall be responsible for assigning the shipment to the appropriate commercial carrier (overnight air shipment is preferred) and for final labeling of the container/cooler. Non-hazardous samples may be hand-delivered to local analytical laboratories by the SMO after meeting all other requirements for packaging and shipping. Refer to LOP 94-03 for detailed sample handling, packaging, and shipping requirements and instructions.

- The SNL/NM Shipping/Receiving Department is responsible for completing the shipping documentation, including the waybill. The SMO shall retain a copy of all sample custody documents including shipping documentation.

Analytical Laboratory Custody Procedure

Sample custody is transferred to the contract laboratory at the time of sample receipt, after which the contract laboratory is responsible for maintenance of unbroken chain-of-custody. The analytical laboratory shall maintain the sample custody records until sample analysis is complete. Sample receipt requirements for the analytical laboratory are:

- At the time of receipt, the subcontract laboratory sample custodian shall sign and date the ARCOC or SARF COC form in indelible ink to acknowledge sample receipt and to accept custody.
- The contract laboratory sample custodian receiving the samples shall verify that the information listed on the ARCOC or SARF COC form is correct and accurately describes the contents of the shipment.
- Maintain records to clearly document all internal transactions as well as the final disposition (e.g., destruction) of the sample.
- Retain samples for at least 60 days, or according to contract or project requirements, after the final analytical report is issued.
- Return custody documentation (original ARCOC, IH SARF, or RPDP SARF or applicable chain-of-custody) to SMO SNL/NM upon completion of analysis.
- Follow the current version of the SMO-SOW

Non-conformance and Corrective Action:

Any non-conformances and corrective actions related to processes described in this procedure and associated corrective actions will be documented, approved, and implemented in accordance with the requirements of the SMO-QAPP and the responsibilities identified in this procedure (Section 4.0). Non-conformances shall be identified by any personnel (e.g., SNL/NM staff; contractor; or contract analytical laboratory).

6.0 RECORDS MANAGEMENT

The SMO shall maintain records to document activities and to provide support for possible evidential proceedings. Records that provide documentary evidence of quality shall be specified, prepared and maintained in accordance with appropriate SNL/NM record-keeping procedures. SMO records shall be transferred to the customer as well as the Records Center for cataloging and storage in accordance with SNL/NM and DOE requirements. The following documentation required by this procedure should be submitted to the Project/Task Leader or SMO personnel for review, approval, and storage in the Records Center:

- ARCOC, IH SARF COC, or RPDP SARF COC Record (hard copy to Records Center)
- Shipper form (hard copy to Records Center)
- Radiation Survey Documentation when applicable (hard copy to Records Center)
- Data package electronic file and EDD
- Nonconformance and corrective action records
- Pertinent correspondence

References

International Air Transport Association (IATA) Dangerous Goods Regulations, current edition, 52nd Edition (updated annually), International Air Transport Association (IATA), Montreal, Canada

National Nuclear Security Administration Service Center Model Statement of Work for Analytical Laboratories, current revision, National Nuclear Security Administration Service Center, Albuquerque, New Mexico.

Sandia National Laboratories/New Mexico Quality Assurance Project Plan for the SNL/NM Sample Management Office (SMO-QAPP), current revision, Sandia National Laboratories/New Mexico Sample Management Office, Albuquerque, New Mexico.

Sandia National Laboratories/New Mexico Sample Handling, Packaging and Shipping Laboratory Operating Procedure (LOP 94-03), current revision, Sandia National Laboratories/New Mexico Sample Management Office, Albuquerque, New Mexico.

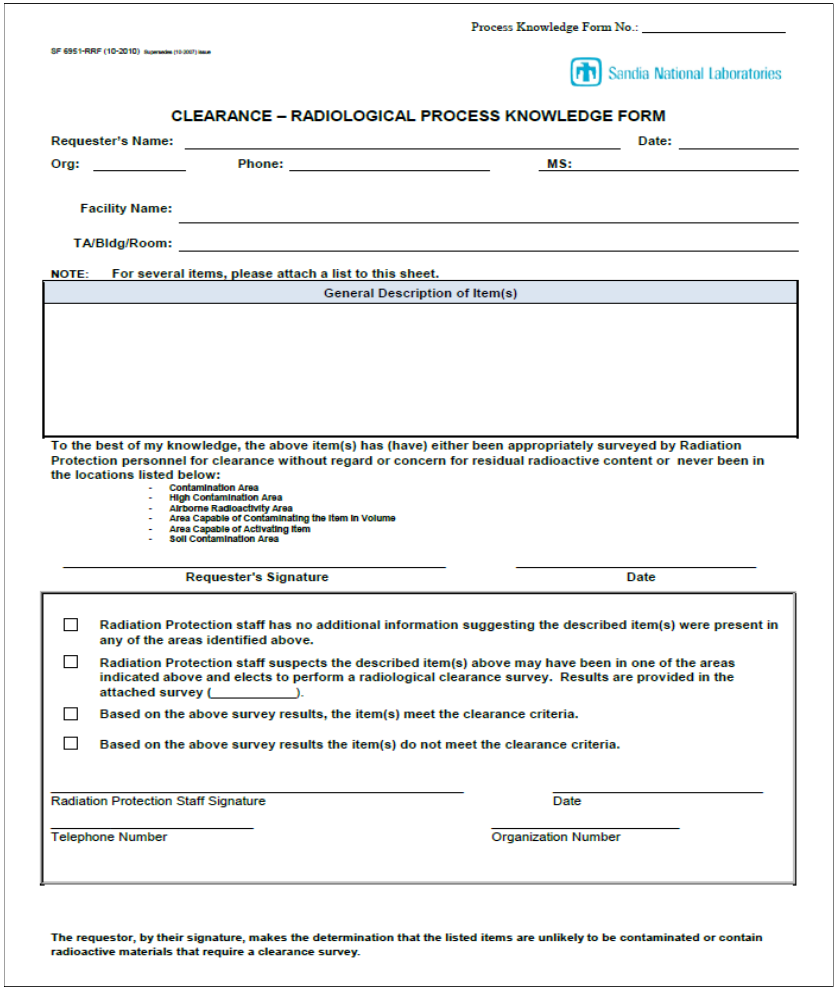
Sandia National Laboratories/New Mexico Statement of Work for Analytical Laboratories, current revision, Sandia National Laboratories/New Mexico Sample Management Office, Albuquerque, NM

U.S. Environmental Protection Agency Code of Federal Regulations (CFR), CFR Title 49, Updated Quarterly as of October 1st, U.S. Environmental Protection Agency, Washington, DC

U.S. Occupational Safety and Health Administration Code of Federal Regulations (CFR), Title 29, Section 1910.1200, Updated Quarterly as of July1st, U.S. Occupational Safety and Health Administration Government Printing Office, Washington, D.C.

ATTACHMENTS

ATTACHMENT A: CLEARANCE RADIOLOGICAL PROCESS KNOWLEDGE FORM SF 6951-RRF



Process Knowledge Form No.: ________________

SF 6951-RRF (10-2010) Supersedes (10-2007) issue

Sandia National Laboratories

CLEARANCE – RADIOLOGICAL PROCESS KNOWLEDGE FORM

Requester's Name: ______________________________ **Date:** ____________

Org: __________ **Phone:** ____________________ **MS:** ______________

Facility Name: ____

TA/Bldg/Room: ____

NOTE: For several items, please attach a list to this sheet.

General Description of Item(s)

To the best of my knowledge, the above item(s) has (have) either been appropriately surveyed by Radiation Protection personnel for clearance without regard or concern for residual radioactive content or never been in the locations listed below:

- Contamination Area
- High Contamination Area
- Airborne Radioactivity Area
- Area Capable of Contaminating the Item in Volume
- Area Capable of Activating Item
- Soil Contamination Area

______________________________ ____________________

Requester's Signature **Date**

☐ **Radiation Protection staff has no additional information suggesting the described item(s) were present in any of the areas identified above.**

☐ **Radiation Protection staff suspects the described item(s) above may have been in one of the areas indicated above and elects to perform a radiological clearance survey. Results are provided in the attached survey (__________).**

☐ **Based on the above survey results, the item(s) meet the clearance criteria.**

☐ **Based on the above survey results the item(s) do not meet the clearance criteria.**

______________________________ ____________________

Radiation Protection Staff Signature Date

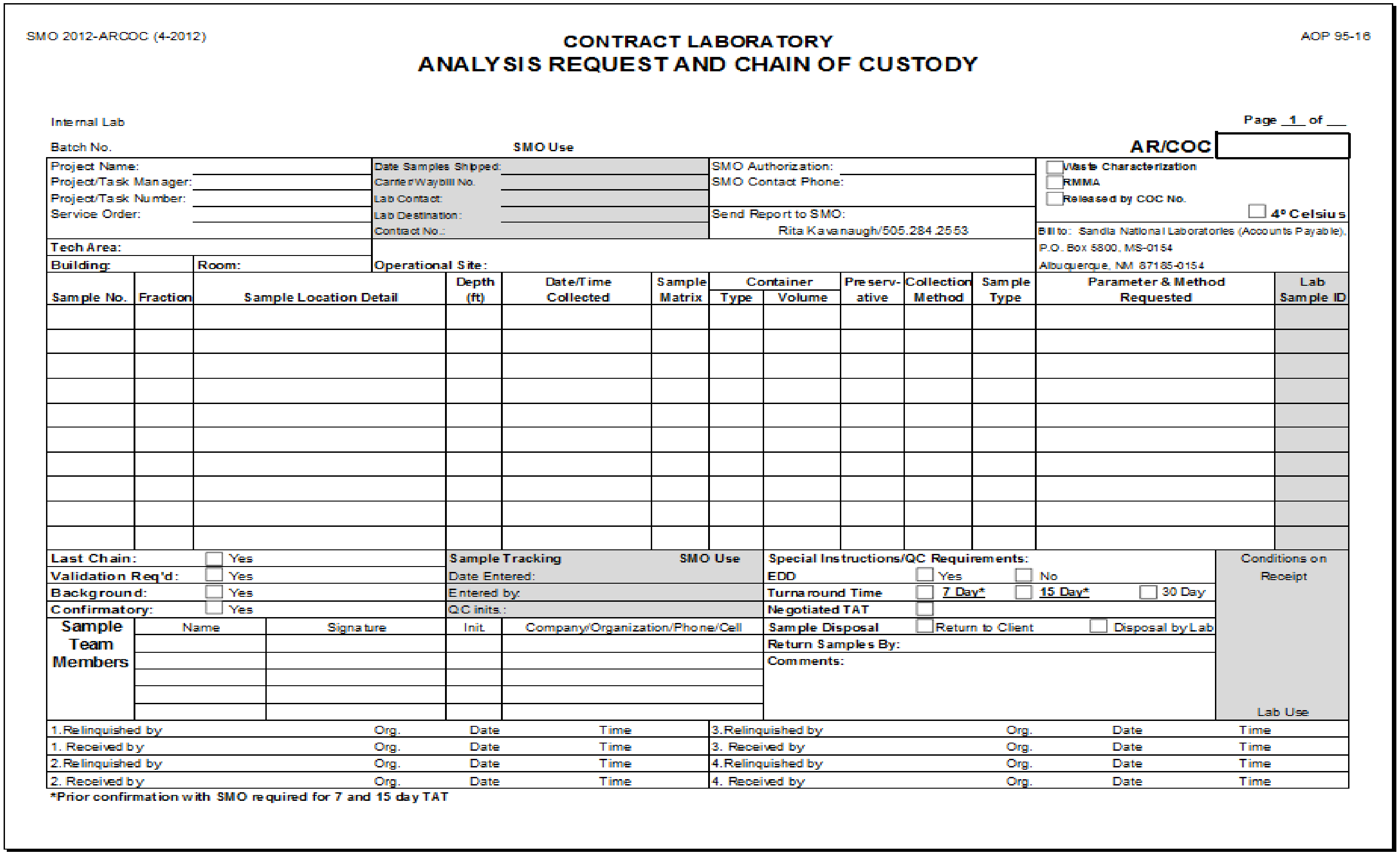
____________________ ____________________

Telephone Number Organization Number

The requestor, by their signature, makes the determination that the listed items are unlikely to be contaminated or contain radioactive materials that require a clearance survey.

ATTACHMENT B:
CONTRACT LABORATORY ANALYSIS REQUEST AND CHAIN-OF-CUSTODY

SMO 2012-ARCOC



SMO 2012-ARCOC (4-2012)

AOP 95-16

CONTRACT LABORATORY
ANALYSIS REQUEST AND CHAIN OF CUSTODY

Page _1_ of ___

Internal Lab
Batch No.

SMO Use

AR/COC ☐

Project Name:
Project/Task Manager:
Project/Task Number:
Service Order:

Date Samples Shipped:
Carrier/Waybill No.
Lab Contact:
Lab Destination:
Contract No.:

SMO Authorization:
SMO Contact Phone:
Send Report to SMO:
Rita Kavanaugh/505.284.2553

☐ Waste Characterization
☐ RMMA
☐ Released by COC No.
☐ **4° Celsius**

Bill to: Sandia National Laboratories (Accounts Payable),
P.O. Box 5800, MS-0154
Albuquerque, NM 87185-0154

Tech Area:
Building: **Room:**
Operational Site:

Sample No.	**Fraction**	**Sample Location Detail**	**Depth (ft)**	**Date/Time Collected**	**Sample Matrix**	**Container**		**Preserv-ative**	**Collection Method**	**Sample Type**	**Parameter & Method Requested**	**Lab Sample ID**
						Type	**Volume**					

Last Chain: ☐ Yes
Validation Req'd: ☐ Yes
Background: ☐ Yes
Confirmatory: ☐ Yes

Sample Tracking **SMO Use**
Date Entered:
Entered by:
QC inits.:

Special Instructions/QC Requirements:
EDD ☐ Yes ☐ No
Turnaround Time ☐ 7 Day* ☐ 15 Day* ☐ 30 Day
Negotiated TAT ☐
Sample Disposal ☐ Return to Client ☐ Disposal by Lab
Return Samples By:
Comments:

Conditions on Receipt
Lab Use

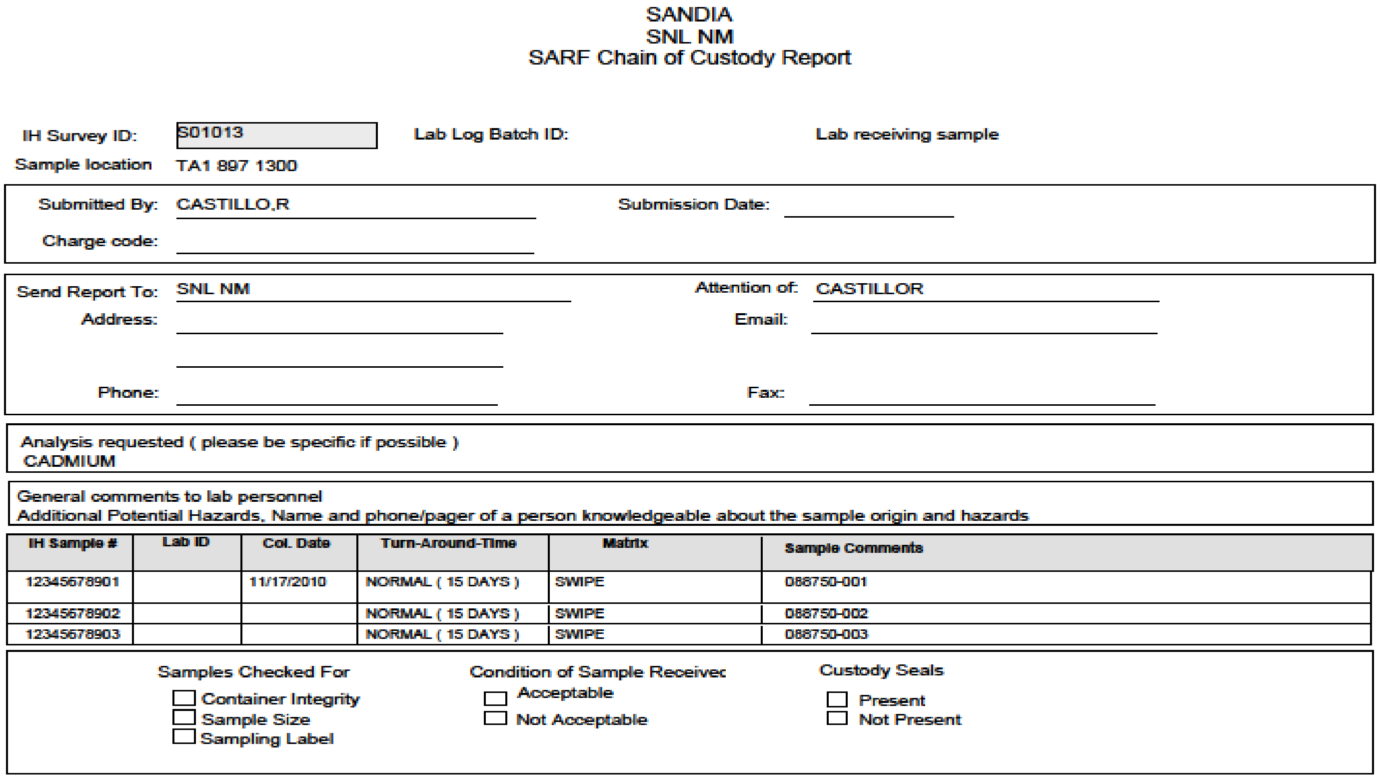
Sample Team Members	Name	Signature	Init.	Company/Organization/Phone/Cell

1.Relinquished by	Org.	Date	Time	3.Relinquished by	Org.	Date	Time
1. Received by	Org.	Date	Time	3. Received by	Org.	Date	Time
2.Relinquished by	Org.	Date	Time	4.Relinquished by	Org.	Date	Time
2. Received by	Org.	Date	Time	4. Received by	Org.	Date	Time

*Prior confirmation with SMO required for 7 and 15 day TAT

This ARCOC is for example purposes only. The ARCOC currently in effect is posted on the 4100 Controlled Documents homepage (SMO 2012-ARCOC).

ATTACHEMNT C: Industrial Hygiene (IH) SARF Chain-of-custody



SANDIA
SNL NM
SARF Chain of Custody Report

IH Survey ID: S01013
Lab Log Batch ID:
Lab receiving sample
Sample location TA1 897 1300

Submitted By: CASTILLO,R
Submission Date:
Charge code:

Send Report To: SNL NM
Attention of: CASTILLOR
Address:
Email:
Phone:
Fax:

Analysis requested (please be specific if possible)
CADMIUM

General comments to lab personnel
Additional Potential Hazards, Name and phone/pager of a person knowledgeable about the sample origin and hazards

IH Sample #	Lab ID	Col. Date	Turn-Around-Time	Matrix	Sample Comments
12345678901		11/17/2010	NORMAL (15 DAYS)	SWIPE	088750-001
12345678902			NORMAL (15 DAYS)	SWIPE	088750-002
12345678903			NORMAL (15 DAYS)	SWIPE	088750-003

Samples Checked For
- ☐ Container Integrity
- ☐ Sample Size
- ☐ Sampling Label

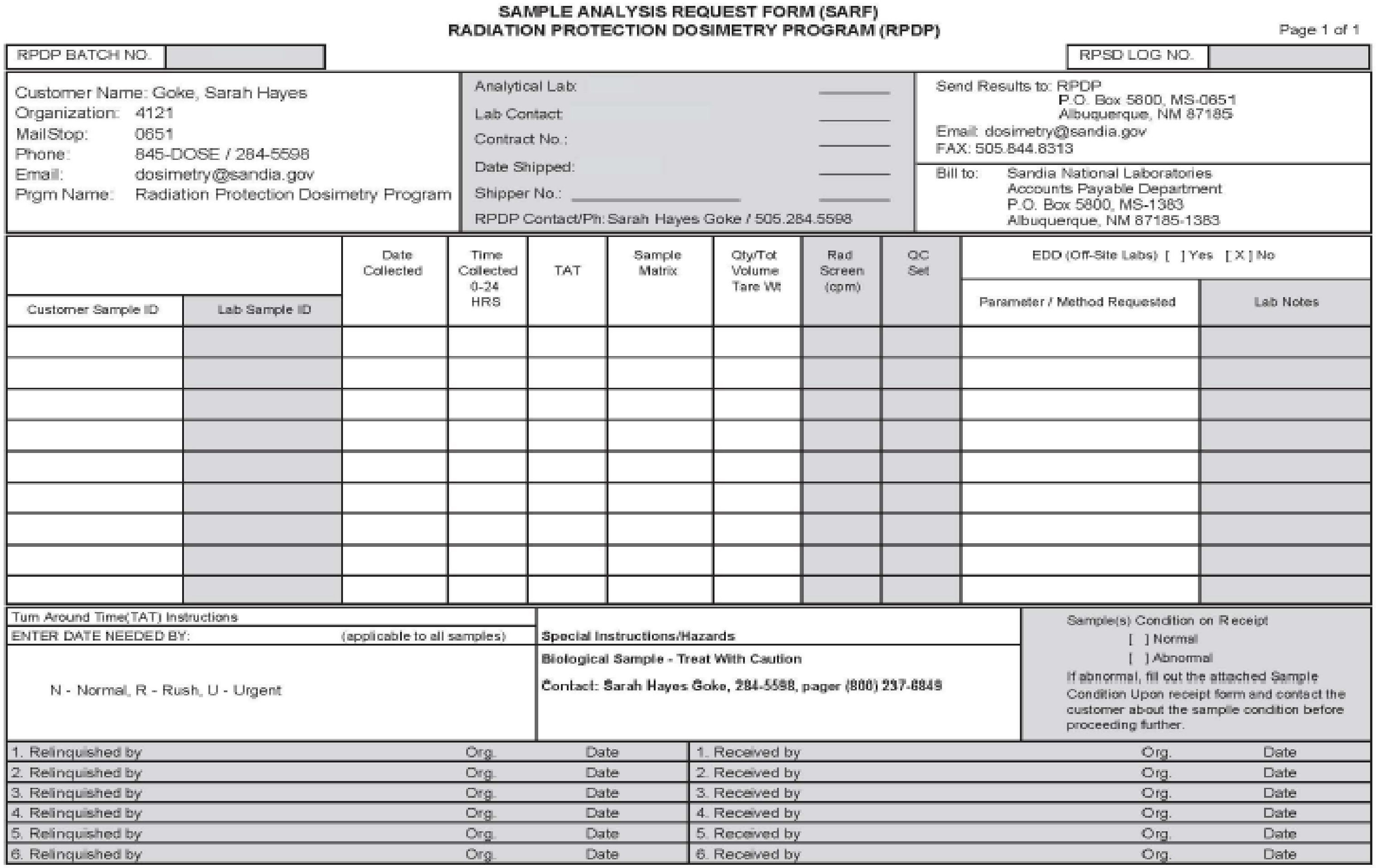
Condition of Sample Receivec
- ☐ Acceptable
- ☐ Not Acceptable

Custody Seals
- ☐ Present
- ☐ Not Present

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.

ATTACHMENT D:
RPDP SAMPLE ANALYSIS REQUEST FORM

RPDP SARF



SAMPLE ANALYSIS REQUEST FORM (SARF)
RADIATION PROTECTION DOSIMETRY PROGRAM (RPDP)

Page 1 of 1

RPDP BATCH NO.

RPSD LOG NO.

Customer Name: Goke, Sarah Hayes
Organization: 4121
MailStop: 0651
Phone: 845-DOSE / 284-5598
Email: dosimetry@sandia.gov
Prgm Name: Radiation Protection Dosimetry Program

Analytical Lab:
Lab Contact:
Contract No.:
Date Shipped:
Shipper No.:
RPDP Contact/Ph: Sarah Hayes Goke / 505.284.5598

Send Results to: RPDP
P.O. Box 5800, MS-0651
Albuquerque, NM 87185
Email: dosimetry@sandia.gov
FAX: 505.844.8313

Bill to: Sandia National Laboratories
Accounts Payable Department
P.O. Box 5800, MS-1383
Albuquerque, NM 87185-1383

EDD (Off-Site Labs) [ ] Yes [X] No

Customer Sample ID	Lab Sample ID	Date Collected	Time Collected 0-24 HRS	TAT	Sample Matrix	Qty/Tot Volume Tare Wt	Rad Screen (cpm)	QC Set	Parameter / Method Requested	Lab Notes

Turn Around Time(TAT) Instructions
ENTER DATE NEEDED BY: (applicable to all samples)
N - Normal, R - Rush, U - Urgent

Special Instructions/Hazards
Biological Sample - Treat With Caution
Contact: Sarah Hayes Goke, 284-5598, pager (800) 237-6849

Sample(s) Condition on Receipt
[ ] Normal
[ ] Abnormal
If abnormal, fill out the attached Sample Condition Upon receipt form and contact the customer about the sample condition before proceeding further.

1. Relinquished by	Org.	Date	1. Received by	Org.	Date
2. Relinquished by	Org.	Date	2. Received by	Org.	Date
3. Relinquished by	Org.	Date	3. Received by	Org.	Date
4. Relinquished by	Org.	Date	4. Received by	Org.	Date
5. Relinquished by	Org.	Date	5. Received by	Org.	Date
6. Relinquished by	Org.	Date	6. Received by	Org.	Date

ATTACHMENT E: ONSITE LABORATORY (RPSD) SAMPLE ANALYSIS REQUEST

RPSD SARF

Sandia National Laboratories
Sample Analysis Programs

Sample Analysis Request Form
Page ____of ____

To be completed by Customer

Shaded areas are for Lab use

Customer Name: ________________
Customer Email ID: ________________
Organization: ________________
Phone: Sample ________________
Location (Bldg/Rm): ________________
Date Results Needed: ________________
Project/Task Number: ________________

Hazards/Special Instructions:

☐ Provide EDD

Batch Log No: ______________
Logged By: ______________

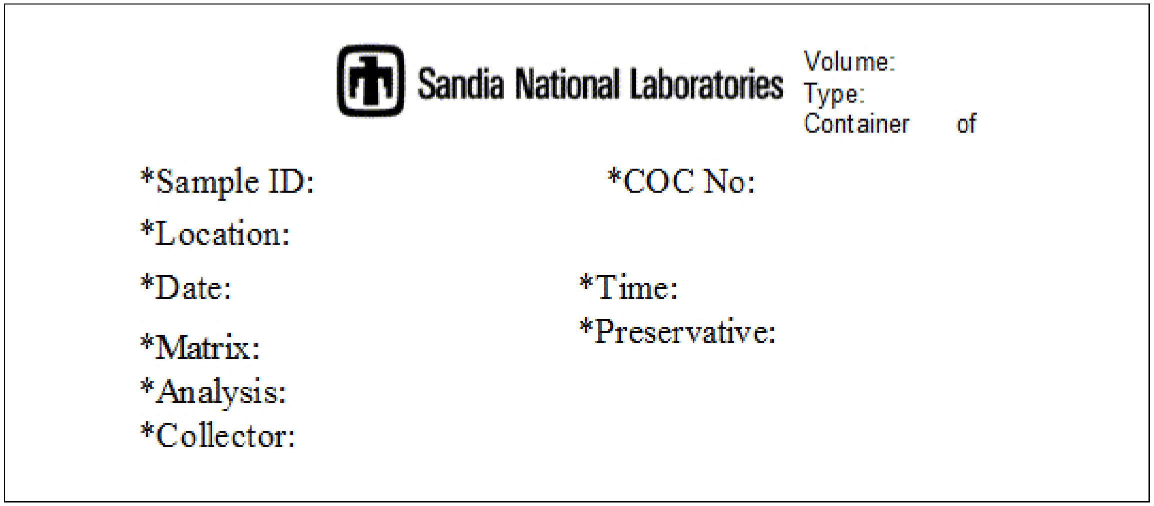
o RPOP - Rad Protection Operation
o RPID - Dosimetry
o RPSD - Sample Diagnostics
o IH - Industrial Hygiene
o DND -DeconDecom
o EXT- External
o SND - Source & Device
o WM – Waste Management
o OTH - Other
o EM - Environment Monitoring
o EMAA - Ambient Air
o EMWW - Waste Water
o EMGW - Ground Water
o EMTS - Terrestrial survell
o EMSW - Storm Water
o CMC – Coop Monitoring Ctr
o ER – Enviroment Restoration

Customer Sample ID	Sample Type	Date/Time Collected	Sample Amount or Flow Rate	Requested Analysis	Survey or COC#	Lab ID	Rad Screen(CPM)	Remarks/Aliquot Amount

Relinquished by ___________________ Date _____________________ Received by ________________________ Date______________________

ATTACHMENT F
SAMPLE LABEL

http://info.sandia.gov/esh/smo/
https://info.sandia.gov/esh/smo_application



Sandia National Laboratories

Volume:
Type:
Container of

*Sample ID:
*COC No:
*Location:
*Date:
*Time:
*Preservative:
*Matrix:
*Analysis:
*Collector:

***Required fields**

ATTACHMENT G
SAMPLE CONTAINERS AND PRESERVATIVES

Holding Times and Sample Preservation

305.1,310.1	Acidity, Alkalinity	Water	Plastic or Glass	4 °C	14 Days	NA
300, 320.1, 325 340.2, 375.X	Bromide, Chloride Fluoride, Sulfate	Water	1 L Plastic	4 °C	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
405	BOD	Water	1 L Plastic	4 °C	48 Hours	NA
9010B, 9014	Total Cyanide	Water	1 L Plastic	4 °C; NaOH; pH > 12	14 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	14 Days	NA
415, 9060	DOC, TOC	Water	250 mL Amber Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
200, 6010, 6020, 7000	All metals except Cr(VI) and Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	180 Days	NA
		Solid/Other	250 mL Glass Jar		180 Days	NA
3060A, 7196A, 7197	Cr(VI)	Water	500 mL Plastic	4 °C	24 Hours	NA
		Solid/Other	250 mL Glass Jar	4 °C	30 Days	NA
245, 7470A, 7471A	Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	28 Days	NA
		Solid/Other	250 mL Glass Jar	4 °C	28 Days	NA
130.1	Hardness	Water		HNO_3 ; pH < 2 4 °C	180 Days	NA
345.1	Iodide	Water		4 °C	24 Hours	NA
300, 353.1, 351, 365	Ammonium, Nitrate + Nitrite Total Phosphorus, TKN	Water	1 L Plastic	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	250 mL Glass Jar	4 °C	28 Days	NA
300, 365, 9210	Nitrate, Nitrate + Nitrite, Nitrite, Ortho Phosphorus	Water	500 mL Plastic	4 °C	48 Hours	NA
		Solid/Other	250 mL Glass Jar	4 °C	48 Hours	NA

1664, 9070	Total Recoverable oil and Grease	Water	1 L Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
376	Sulfide	Water	1 L Glass	4 °C; NaOH; Zinc acetate; pH > 9	7 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	7 Days	NA
160	TDS, TSS, TS	Water	1 L Plastic	4 °C	7 Days	NA
9020B	TOX	Water	1 L Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
418.1, 8440	TPH	Water	1 L Amber Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
1664		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
9065, 9066	Total Recoverable Phenols	Water	1 L Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid	125 mL Glass Jar	4 °C	28 Days	NA
150, 9040B	pH	Water	125 mL Plastic	4 °C	24 Hours	NA
110, 180	Color, Turbidity	Water	500 mL Plastic	4 °C	48 Hours	NA
120, 9050	Specific Conductance	Water	125 mL Plastic	4 °C	28 Days	NA
All radiochemical parameters		Water	1 L Plastic (2 x 2 L Preferred)	HNO_3 ; pH < 2	180 Days	NA
except Rn-222 and tritium		Solid/Other	250 mL Glass Jar		180 Days	NA
Rn-222	Radon 222	Water	3 x 40 mL Amber Glass Vial	None	72 Hours	NA
Tritium	^3H	Water	1 L Glass		180 Days	NA
		Solid/Other	Required sample size will vary with solid moisture content			NA
8015	Petroleum Hydrocarbons	Water	2 x 1 L Amber Glass Bottle	4 °C	14 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	14 Days	40 Days

8021B	Halogenated Volatile Organics	Water	3 x 40 mL Amber Glass Vial	4 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	4 °C	14 Days	NA
8081, 8082	Organochlorine Pesticides, PCBs	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
8141A	Organophosphorous Compounds	Water	4 L Amber Glass Bottle	4 °C; NaOH or H_2SO_4; pH 5-8	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
8151	Chlorinated Herbicides	Water	4 L Amber Glass Bottle	4 °C;	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
8260B (Modified)	Volatile Organics by GC-MS	Water	3 x 40 mL Amber Glass Vial	4 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	4 °C	14 Days	NA
8270C	Semi-volatile Organics by GC-MS	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
8280A	Polychlorinated Dioxins and Furans by GC/MS	Water	4 L Amber Glass Bottle	4 °C	30 Days	45 Days
		Soil/Other	250 Glass Jar	4 °C	30 Days	45 Days
8318	N-Methylcarbamate Pesticides by HPLC	Water	4 L Amber Glass Bottle	4 °C; 0.1 N $ClCH_2CO_2H$, pH 4 - 5	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	7 Days	40 Days
8330	Nitroaromatics and Nitramines by HPLC	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
TO-13	PAHs in Filter Cartridges	PUF, Tenax, or XAD-2 Filter Cartridge		4 °C	7 Days	40 Days
TO-14/ TO-15	VOC in Air	SUMMA® Canister			30 Days (by consensus)	
305.1,310.1	Acidity, Alkalinity	Water	Plastic or Glass	4 °C	14 Days	NA
300, 320.1, 325 340.2, 375.X	Bromide, Chloride Fluoride, Sulfate	Water	1 L Plastic	4 °C	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
405	BOD	Water	1 L Plastic	4 °C	48 Hours	NA

9010B, 9014	Total Cyanide	Water	1 L Plastic	4 °C; NaOH; pH > 12	14 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	14 Days	NA
415, 9060	DOC, TOC	Water	250 mL Amber Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
200, 6010, 6020, 7000	All metals except Cr(VI) and Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	180 Days	NA
		Solid/Other	250 mL Glass Jar		180 Days	NA
3060A, 7196A, 7197	Cr(VI)	Water	500 mL Plastic	4 °C	24 Hours	NA
		Solid/Other	250 mL Glass Jar	4 °C	30 Days	NA
245, 7470A, 7471A	Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	28 Days	NA
		Solid/Other	250 mL Glass Jar	4 °C	28 Days	NA
130.1	Hardness	Water		HNO_3 ; pH < 2 4 °C	180 Days	NA
345.1	Iodide	Water		4 °C	24 Hours	NA
300, 353.1, 351, 365	Ammonium, Nitrate + Nitrite Total Phosphorus, TKN	Water	1 L Plastic	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	250 mL Glass Jar	4 °C	28 Days	NA
300, 365, 9210	Nitrate, Nitrate + Nitrite, Nitrite, Ortho Phosphorus	Water	500 mL Plastic	4 °C	48 Hours	NA
		Solid/Other	250 mL Glass Jar	4 °C	48 Hours	NA
1664, 9070	Total Recoverable oil and Grease	Water	1 L Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
376	Sulfide	Water	1 L Glass	4 °C; NaOH; Zinc acetate; pH > 9	7 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	7 Days	NA
160	TDS, TSS, TS	Water	1 L Plastic	4 °C	7 Days	NA
9020B	TOX	Water	1 L Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
418.1, 8440 1664	TPH	Water	1 L Amber Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA

9065, 9066	Total Recoverable Phenols	Water	1 L Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid	125 mL Glass Jar	4 °C	28 Days	NA
150, 9040B	pH	Water	125 mL Plastic	4 °C	24 Hours	NA
110, 180	Color, Turbidity	Water	500 mL Plastic	4 °C	48 Hours	NA
120, 9050	Specific Conductance	Water	125 mL Plastic	4 °C	28 Days	NA
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
Rn-222	Radon 222	Water	3 x 40 mL Amber Glass Vial	None	72 Hours	NA
Tritium	^3H	Water	1 L Glass		180 Days	NA
		Solid/Other	Required sample size will vary with solid moisture content			NA
8015	Petroleum Hydrocarbons	Water	2 x 1 L Amber Glass Bottle	4 °C	14 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	14 Days	40 Days
8021B	Halogenated Volatile Organics	Water	3 x 40 mL Amber Glass Vial	4 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	4 °C	14 Days	NA
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8141A	Organophosphorous Compounds	Water	4 L Amber Glass Bottle	4 °C; NaOH or H_2SO_4; pH 5-8	7 Days	40 Days
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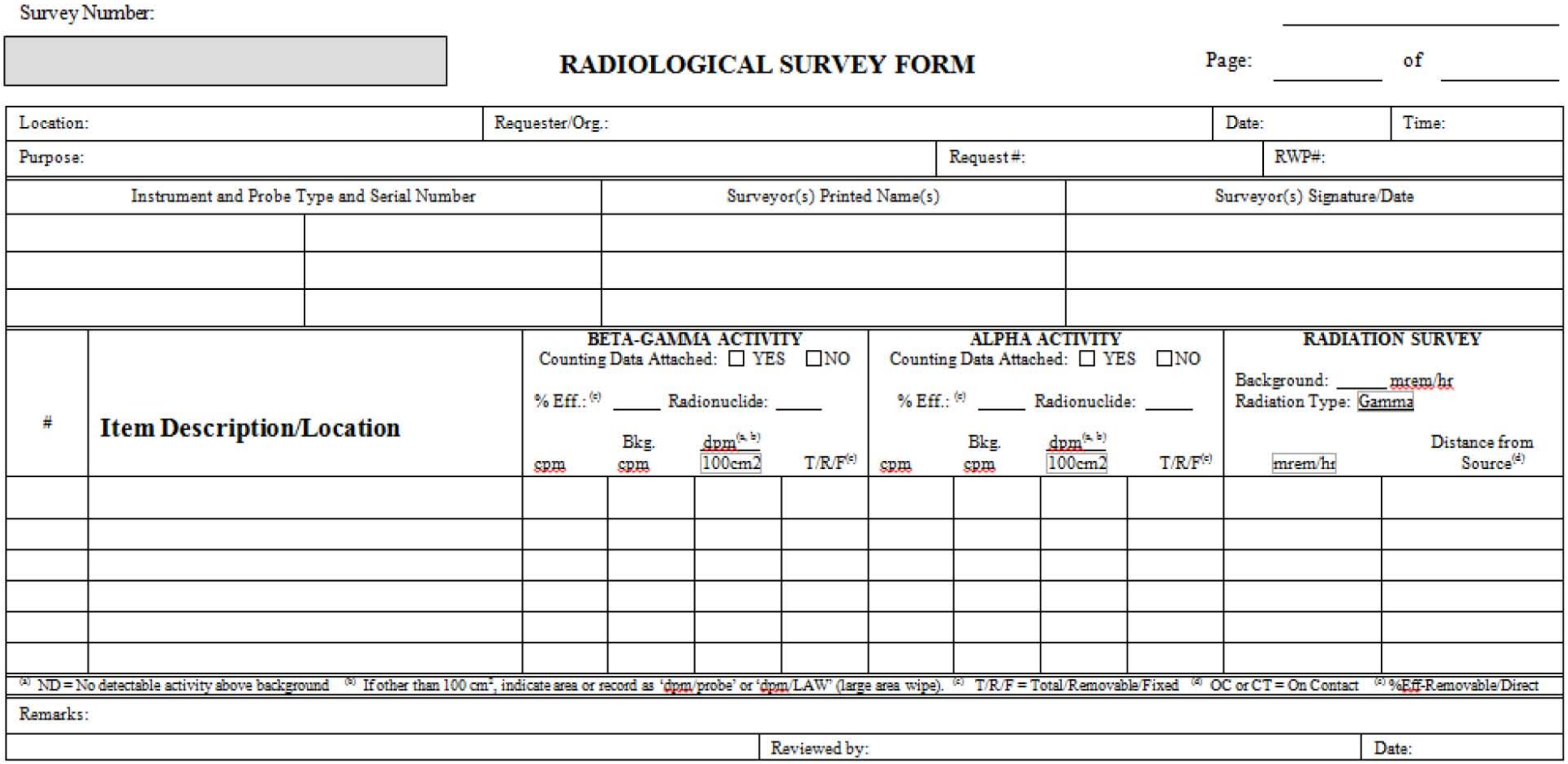
8151	Chlorinated Herbicides	Water	4 L Amber Glass Bottle	4 °C;	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
8260B (Modified)	Volatile Organics by GC-MS	Water	3 x 40 mL Amber Glass Vial	4 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	4 °C	14 Days	NA

8270C	Semi-volatile Organics by GC-MS	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
8280A	Polychlorinated Dioxins and Furans by GC/MS	Water	4 L Amber Glass Bottle	4 °C	30 Days	45 Days
		Soil/Other	250 Glass Jar	4 °C	30 Days	45 Days
8318	N-Methylcarbamate Pesticides by HPLC	Water	4 L Amber Glass Bottle	4 °C; $0.1\ \underline{N}\ ClCH_2CO_2H$, pH 4 - 5	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	7 Days	40 Days
8330	Nitroaromatics and Nitramines by HPLC	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	4 °C	14 Days	40 Days
TO-13	PAHs in Filter Cartridges	PUF, Tenax, or XAD-2 Filter Cartridge		4 °C	7 Days	40 Days
TO-14						

Attachment H: Radiological Survey Form

http://www-irn.sandia.gov/esh/radpro_procedures/forms/rsf.dot



Survey Number:

RADIOLOGICAL SURVEY FORM

Page: ______ of ______

Location:	Requester/Org.:	Date:	Time:
Purpose:	Request #:	RWP#:	

Instrument and Probe Type and Serial Number		Surveyor(s) Printed Name(s)	Surveyor(s) Signature/Date

#	Item Description/Location	BETA-GAMMA ACTIVITY Counting Data Attached: ☐ YES ☐ NO % Eff.: [e] ____ Radionuclide: ____				ALPHA ACTIVITY Counting Data Attached: ☐ YES ☐ NO % Eff.: [e] ____ Radionuclide: ____				RADIATION SURVEY Background: ____ mrem/hr Radiation Type: Gamma	
		cpm	Bkg. cpm	dpm[a, b] / 100cm2	T/R/F[c]	cpm	Bkg. cpm	dpm[a, b] / 100cm2	T/R/F[c]	mrem/hr	Distance from Source[d]

[a] ND = No detectable activity above background [b] If other than 100 cm^2, indicate area or record as 'dpm/probe' or 'dpm/LAW' (large area wipe). [c] T/R/F = Total/Removable/Fixed [d] OC or CT = On Contact [e] %Eff-Removable/Direct

Remarks:

Reviewed by: 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.

Page _____ of ______

AUTHORIZED USERS LIST

Document Title: Sample Management and Custody

Administrative Operating Procedure

Document Number: AOP 95-16 **Revision:** 05

By my signature below, I affirm that I have read and understand this document, and all references called out in procedural steps, and I agree to operate within the stated constraints.

Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date

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SANDIA NATIONAL LABORATORIES
LONG-TERM STEWARDSHIP

FIELD OPERATING PROCEDURE

FOP 05-01

GROUNDWATER MONITORING WELL SAMPLING AND FIELD ANALYTICAL MEASUREMENTS

Author: ______ **Date:** 1/10/12
Robert Ziock, Subject Matter Expert

Approved: ______ **Date:** 10 Jan 12
Michael Skelly, Project Leader

Approved: ______ **Date:** 1/10/12
Don Schofield, Field Support Operations Project Leader

Approved: Pamela Puissant **Date:** 1/24/12
Pamela Puissant, Department Manager

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*]	*No*

EFFECTIVE DATE: 1/24/12

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Revision History

Revision	Review Date	Effective Date	Summary of Changes
1	9/29/2005	10/10/2005	New document
2	6/13/2007	8/16/2007	Formatting changes. Updated section 2, Roles and Responsibilities; section 3, Training Qualifications; section 9, References.
3	11/12/2009	11/12/2009	Formatting changes. Added work planning and control information to section 4.0, Health & Safety. Updated section 3, Training Qualifications; section 9, References.
4	1/10/2012	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 9, References.

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Table of Contents

TABLES

ATTACHMENTS

FORMS

ARCOC – Sample Management Office – Analysis Request and Chain of Custody Form
EP 2009-ALW – Environmental Planning – Activity Level Work Evaluation Form
EP 2009-AUL – Environmental Planning – Authorized Users List
EP 2009-OJT – Environmental Planning – On-the-Job Training
EP 2009-WRA – Environmental Planning – Work Resumption Authorization Form

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Acronyms & Abbreviations

AOP	administrative operating procedure
ARCOC	analysis request and chain of custody
DI	deionized
DO	dissolved oxygen
EDMS	Environmental Data Management System
ES&H	Environment, Safety and Health
FOP	field operating procedure
gal/ft^3	gallon(s)/cubic foot
ft	foot/feet
in	inch(es)
LOP	laboratory operating procedure
LTES	Long Term Environmental Stewardship
mL	milliliter(s)
min	minutes(s)
OJT	on-the-job training
ORP	oxidation/reduction potential
OSHA	Occupational Safety and Health Administration
pH	potential for hydrogen
PHS	primary hazard screening
PLA	plan
RCRA	Resource Conservation and Recovery Act
SAP	sampling and analysis plan
SC	specific conductance
SMO	Sample Management Office
SNL/NM	Sandia National Laboratories, New Mexico
THA	task hazard analysis
TOC	total organic carbon
VOC	volatile organic compound

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1.0 PURPOSE, SCOPE, AND OWNERSHIP

Purpose This field operating procedure (FOP) provides instruction on collecting a groundwater sample from a monitoring well that is representative of in situ groundwater conditions and is suitable for laboratory analysis. The sample must be conducted per regulatory requirements and established agency guidance to provide legally defensible analytical data for regulatory compliance.

Scope The scope of this procedure is limited to well purging, field analytical measurements, and the collection of samples from monitoring wells as part of Sandia National Laboratories, New Mexico (SNL/NM) groundwater monitoring activities.

Ownership The Long-Term Stewardship Department is responsible for development, approval, distribution, revision, and control of this document.

2.0 ROLES AND RESPONSIBILITIES

The **project leader** is responsible for the following:

- Reviewing and recommending approval of this procedure.
- Providing overall coordination and management of the project.
- Providing a sampling and analysis plan (SAP) that meets prescribed regulatory or programmatic requirements.
- Reviewing completed field forms and data pertaining to the sampling activities.
- Reporting all information as may be 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. It details sampling activities for the field support operations project leader and field technicians. It summarizes sampling procedures, analytical parameters, field measured parameters, purge requirements, and waste management tasks. It also identifies monitoring well characteristics that may extend the sampling period (e.g., low yield wells, well construction issues, etc.).
- Coordinating sampling activities with the SNL/NM lead investigators, regulators, Kirtland Air Force Base and their environmental contractors, Sample Management Office (SMO), and field technicians.
- Providing the field support operations project leader with a copy of the mini-SAP.
- Coordinating sampling procedure variances with the project leader and the field support operations project leader.
- Reviewing completed field forms and data pertaining to the sampling activities.
- Ensuring that all data quality requirements are performed.
- Providing the project leader with a summary of field activities and sampling results.

- Reporting all information as may be required by regulations or directives.
- Submitting completed field forms to the Customer Funded Record Center and entry of relevant data to the Environmental Data Management System (EDMS) database.
- Managing, coordinating, and disposing of purge water and other waste generated from field operations in compliance with SNL/NM Corporate Policy ESH100 Environment Safety & Health, and FOP 05-04, *Groundwater Waste Management Plan.*
- Reviewing and providing recommendations for revisions to this procedure (if necessary).

The **field support operations project 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 for the on-the-job training (OJT) of new field technicians.
- Assigning field technicians (qualified by training and experience) to conduct the activities described in this procedure.
- Coordinating sampling activities with the sampling coordinator, SMO, and field technicians.
- Ensuring the materials and equipment necessary to perform the work are available.
- Providing the field technicians with a copy of the mini-SAP.
- Reviewing field documentation.
- Maintaining the calibration records.
- Maintenance of the training matrix for all field personnel.
- Maintaining, reviewing, and revising all technical work documents.
- Notifying the project leader and sampling coordinator of unusual field conditions, wells requiring maintenance, or breach of well security.
- Reviewing and providing recommendations for revisions to this procedure (if necessary).

The **field technician** is responsible for the following:

- Completing all necessary and required training as specified by the field support operations project leader.
- Maintaining and decontaminating equipment.
- Field checking instruments according to the manufacturer's instructions and this procedure.
- Purging the well in preparation for sample collection.
- Collecting and recording field measurements as required by this procedure and the mini-SAP.
- Collecting, preserving, and storing samples as specified in the mini-SAP and in accordance with administrative operating plan (AOP) AOP 95-16, *Sample Management and Custody*.
- Delivering samples to SMO packaging and shipping facility within the required holding times.
- Managing purge water and other generated waste.

- Securing wells.
- Informing the field support operations project leader of wells requiring maintenance or breach of well security.
- Completing and reviewing field documentation forms.
- Providing recommendations for revisions to this procedure (if necessary).

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 Environmental Stewardship (LTES) Groundwater Monitoring Activities*.
- OJT for new field personnel performing groundwater sampling activities. Document training by completing On-the-Job Training form (EP 2009-OJT).
- Read and sign AOP 95-16, *Sample Management and Custody*.
- Read and sign FOP 03-02, *LTES Groundwater Level Data Acquisition and Management*.
- Read and sign laboratory operating procedure (LOP) LOP 94-03, *Sample Handling, Packaging and Shipping*.
- 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 Plan (PLA) PLA 05-09, *Groundwater Monitoring 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

Table 1. Training Course List (concluded)

Course Code	Course Title
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
RAD230	Radiological Worker II Training
RAD230R	Radiological Worker II Retraining
RGH100	Crane, Rigging, Hoisting, & Hands-on Training

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 HEALTH AND SAFETY

A task hazard analysis (THA) has been performed on the activities described in this FOP as well as a hazard assessment survey performed by a SNL/NM industrial hygienist. They are detailed in the PLA 05-09, *Groundwater Sampling Health and Safety Plan*. The THA was performed in conjunction with PHS SNL05A01241, *LTES Groundwater Monitoring Activities*.

An Activity Level Work Evaluation Form (EP 2009-ALW) was completed and approved by the department manager as required by AOP 09-10, *Work Planning and Control*.

A field technician or the field support operations project leader shall conduct a tailgate safety meeting and fill out a Tailgate Safety Meeting Form prior to the start of groundwater sampling activities as described in PLA 05-09, *Groundwater Sampling Health and Safety Plan*.

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 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.

5.0 DEFINITIONS

analysis request and chain of custody (ARCOC) – Method for documenting the history and possession of a sample from the time of its collection, through its analysis and data reporting, to its final disposition.
decontamination – A process used to clean equipment.

dedicated sampling equipment – Sampling equipment that is installed for use in only one monitoring well to prevent potential cross-contamination between wells.

duplicate (or split) sample **–** A sample which is split into subsamples contemporaneous in time and space.

equipment blank – A sample collected from chemically pure water (reagent-grade deionized [DI] water) that is passed through an item of field sampling equipment and returned to the laboratory for analysis. It is used to determine the effectiveness of equipment decontamination process. This may also be referred to as a decontamination rinsate blank.

field blank – Chemically pure water (reagent-grade DI water) that is subjected to all aspects of sample collection, field processing, preservation, transportation, and laboratory handling as an environmental sample.

groundwater – Water beneath the land surface contained in interconnected pores or secondary openings in the saturated zone that is under hydrostatic pressure. The water that enters wells and issues from springs.

headspace – The empty volume in a sample container between the water level and the cap.

mini-SAP – A detailed document describing the location of wells to be sampled; the frequency of sampling; the analysis to be performed; purge volumes, sample containers, sample preservatives, and sample holding times; and the analytical methods to be utilized. The plan references procedures used to collect, handle, and analyze groundwater samples. It includes all quality control measures that will be implemented to ensure that all activities meet any prescribed regulatory or programmatic requirements.

monitoring well – A well that is constructed for the purpose of extracting groundwater for physical, chemical, or biological testing, or for measuring water levels.

preservation – Storage conditions or addition of a reagent that will minimize the change in concentration of a constituent of interest until analysis can be performed.

purge – The process of removing stagnant water from a well prior to sampling.

Sample Management Office (SMO) – A SNL/NM organization responsible for coordinating with analytical laboratories to have groundwater samples analyzed. SMO receives sample bottles from the laboratory and is responsible for shipping samples.
saturated casing volume – The volume of water contained in one length of the saturated screen casing plus the volume of water contained in the borehole annulus surrounding the outside of the screen casing interval.

static water level – The elevation of the top of a column of water in a monitoring well that is at equilibrium.

trip blank – Laboratory prepared samples of chemically pure water (reagent-grade DI water) used to assess volatile organic compound contamination of the sample container, laboratory preparation water, or contamination resulting from handling procedures. Trip blanks are prepared in the same type of bottle used for an environmental sample and is kept with the set of sample bottles both before and after sample collection.

volatile organic compounds (VOCs) – Liquids or solid organic compounds that exhibit a tendency to pass into the vapor state.

6.0 EQUIPMENT AND MATERIALS

Two pumping methods are used to collect groundwater samples. Each method uses an inert gas (nitrogen) to operate the pump. No aeration of the sample occurs because the sample is isolated from the driving gas. The pump methods are:

1. A portable non-dedicated piston pump (Bennett model 1800 or equivalent) is used to purge water from the well casing.

2. A dedicated pneumatic bladder pump (QED micropurge) is used for low-flow technique that uses a dedicated pneumatic bladder sampling pump (QED micropurge). Only the discharge line from the pump must be purged.

The following list includes equipment and materials necessary to collect a sample, document its collection, and request the laboratory analyses. Not all of the equipment may be required for each sampling event. The list depends on the methods used for purging and sampling, and the potential presence of contaminants in groundwater at the site. Details are specified in the mini-SAP.

<u>Consumables</u>

- potential for hydrogen (pH) paper.
- DI water.

- Decontamination supplies listed in FOP 05-03 (detergents, DI water, reagent grade nitric acid, buckets, brushes, etc.). Additional decontamination requirements and supplies may be specified in the mini-SAP.
- Small and large zip-lock and bubble wrap bags.
- Insulated ice chest (coolers), blue ice or ice.
- Duct tape.
- Paper wipes.
- Wash bottles.
- Sample containers with the required reagents for field preservation of samples. They are identified in the mini-SAP and are provided by SMO and/or the laboratory. (Note: Visually inspect preservatives for discoloration or degradation prior to use to ensure reagent quality.).
- Seals (custody seal tape).
- Indelible black ink pens.
- Personal protective equipment. Reference PLA 05-09, *Groundwater Sampling Health and Safety Plan*.

Documentation Forms

- Groundwater Sample Collection Field Equipment Check Log, (FOP 05-02, Attachment A).
- Field Measurement Log for Groundwater Sample Collection (Attachment A).
- Examples of Sample Label and Sample Identification Numbers (Attachment B).
- Analysis request and chain of custody (ARCOC).

Other Equipment

- Portable Bennett piston pump system or equivalent.
- Compressed nitrogen gas cylinders with associated regulators, calibrated pressure relief valves and high pressure air lines, as necessary, to operate portable Bennett piston pump system or equivalent.
- Water level indicator.
- Empty 55-gallon close head poly drums for purge water/decontamination fluids.
- 0.45 micron membrane filter for filtering the dissolved metals.
- Equipment for groundwater field parameters (pH, specific conductivity [SC], temperature, dissolved oxygen [DO], turbidity, oxidation/reduction potential [ORP]; calibration standards) as specified in FOP 05-02.
- Equipment for field analysis water chemistry such as a digital titrator and colorimeter.

Additional Items

- Calculator.
- Location map of monitoring wells to be sampled.
- Well construction details of monitoring wells to be sampled (depth from top of casing, casing diameters, screen length and depths).

- Key to well lock(s) and for gate access to site (if applicable).
- Equipment manuals.
- Tool box.
- 2-way radio.

7.0 PROCEDURE

7.1 Preparation

The field operations project leader and/or field technician must complete the following prior to field operations:

1. Review the mini-SAP with the sampling coordinator and schedule the sampling dates.

2. Obtain the required information listed below:

    a. Location of wells.
    b. Depth of wells.
    c. Diameter of well casings and other well construction data.
    d. Method to be used to purge wells.
    e. Methods used to sample wells.
    f. Analytical parameters including number of equipment blanks, duplicate samples, trip blanks.

3. Coordinate site access with the appropriate organization.

4. Notify SMO of expected sampling and delivery dates, number of samples, required analyses, and turnaround time for analytical results. Obtain the sample containers from SMO. Sample containers must be prepared with the appropriate preservatives by the laboratory or SMO prior to sampling.

5. Make provisions for proper storage and disposal of purge water, decontamination fluids, and other wastes generated during purging and sampling activities.

6. Obtain sample identification numbers from the SMO or field support operations project leader and prepare sample labels. Affix labels to sample bottles.

7. Complete as much as possible of the following forms prior to field operations:

    a. Groundwater Sample Collection Field Equipment Check Log (FOP 05-02, Attachment A),
    b. Field Measurement Log for Groundwater Sample Collection (Attachment A),
    c. ARCOC,

8. Load and secure the equipment and supplies in the sampling vehicle.

7.2 Documentation

Document all sampling activities by completing the appropriate forms. Use a dark indelible ink pen. Make any necessary corrections by drawing a single line through the incorrect entry, initial and date it, and place the correct entry adjacent to the incorrect entry.
The documentation forms are briefly discussed below:

Field Measurement Log for Groundwater Sample Collection, (Attachment A)**.** This form is used to record the following:

- Water level depth
- Purge volumes
- Purge field parameters
- Instrument calibration
- Sample collection
- Well condition
- Weather condition

Sample Label, (Attachment B). An adhesive-backed waterproof label is affixed to each sample container before sample collection. Complete the following entries on the label with a black waterproof ink pen:

- Sample ID #
- Sample Type
- Analysis
- Location
- Date
- Time
- Preservative
- Collector

See AOP 95-16 for more information on the sample label.

Sample Identification Number (Sample # / Sample ID), (Attachment B). A sample consists of groundwater collected at a specific location and time. A unique sample number and sample identification is assigned to each sample collected. The sample number is placed in the appropriate space of the Sample Label, and the ARCOC record.

An example of a Sample # is **001234-001** where the

- **001234 –** References a unique sample identification number. This number is obtained by going to the SMO Home page and clicking the “Control Numbers” tab.
- **-001 –** A fraction that references an analysis. For example -001 for a VOC analysis, -017 for a major cations analysis, and -027 for a cyanide analysis.

An example of a Sample Identification # is **MWL-MW1** where the

- **MWL –** References the monitor well location.
- **MW1 –** References the monitor well number.

See AOP 95-16 for more information on the Sample Identification Number.

***Analysis Request and Chain-of-Custody Record* (ARCOC).** An original ARCOC record must accompany all samples from the field to the laboratory. The ARCOC provides a legal record of the sample from collection to disposal. The ARCOC also specifies the sample analysis. Instructions for completing the ARCOC are provided on the back of the form. ARCOC numbers are obtained at the SMO Home page and clicking the “Control Numbers” tab.

See AOP 95-16 for more information on the ARCOC.

7.3 Decontamination

Decontaminate all equipment used to sample groundwater prior to placement in a well. Follow the directions listed in FOP 05-03 for decontaminating equipment.

7.4 Static Water Level Measurements

Water level information is used to calculate the volume of water in a well casing and the minimum amount required for purging. The static water level is measured in each well prior to purging or obtaining a sample. Static water level measurements are taken to the nearest 0.01 foot using a water level indicator. Measurements are referenced to a surveyed mark of known elevation at the top the well casing. See FOP 03-02 for collecting a static water level measurement. (Note: Because the static water level measurement is used to calculate a purge volume it is not necessary to follow every step of FOP 03-02. Use it only as general guidance for obtaining a static water level measurement.)

7.5 Well Evacuation (Purging)

Purging a well removes stagnant water so that a representative sample of the groundwater can be obtained. Purging requirements are affected by one or more of the following and are detailed in the mini-SAP:

- Permit requirements
- The pump and sample method used (conventional vs. low-flow)
- Well construction
- Well recharge rate

Purge the well at a rate that creates minimal disturbance to the groundwater flow regime. This ensures the most representative sample of the groundwater. Monitor the water level during the purging process to help indicate minimal disturbance. Purge the well until the volume specified in the mini-SAP is met and until groundwater field parameters stabilize. See section 7.6 for more information on measuring groundwater field parameters. Instructions for purging using the two different pump methods are listed in the next two sections.

7.5.1 Portable Non-Dedicated Piston Pump System

Install the pump according to its operating manual and criteria specified in the mini-SAP. The pump intake should be se at or near the bottom of a well's screened interval. Nitrogen gas is used to operate the pump. The flow rate is controlled by varying the gas pressure on the pump. A minimum of one saturated casing volume (unless otherwise specified in the mini-SAP) needs to be purged when using a portable non-dedicated piston pump system. A saturated casing volume is defined as the volume of one length of the saturated screen ($V1$) plus the borehole annulus surrounding the outside of the well screen interval ($V3$). The following formulas are used to calculate a saturated casing volume:

$V1 = R1^2 \text{ x } \pi \text{ x } (H2 - H1) \text{ x } 7.48$ gallon/cubic feet (gal/ft^3)
$V2 = R2^2 \text{ x } \pi \text{ x } (H2 - H1) \text{ x } 7.48$ gal/ft^3
$V3 = (V2 - V1) \text{ x } 0.30$

Minimum purge volume or saturated casing volume = $V3 + V1$

where:

π = 3.14
$R1$ = radius of the well casing feet (ft) = [diameter of well casing inch (in) x 1(ft)/12 (in)] / 2
$R2$ = radius of borehole (ft) = [diameter of borehole (in) x 1 (ft)/12 (in)] / 2
$V1$ = volume of submerged screen length casing interval
$V2$ = volume of submerged screen length annulus
$V3$ = submerged annulus volume
$H1$ = depth to water in screen (ft) not to exceed length of screen
$H2$ = well depth (ft)
$H2 - H1$ = static water height (ft) in well screen
0.30 = filter pack porosity

Begin purging the well after the minimum purge volume has been determined. If possible, purge the well at a flow rate equal to the recharge rate of the well. A pumping rate that is equal to the recharge rate will keep the water level in the well constant. Otherwise, turn down the flow rate to the lowest level possible and still maintain flow at the surface. Record groundwater field parameters at a minimum of every 5 gallons. Groundwater field parameters must stabilize before samples are collected. See Section 7.6 for more information on collecting groundwater field parameter data.

If the well goes dry during the purging process, allow the water in the well to recover to 80 percent of the static water level. If the recovery time exceeds 2 hours, collect the samples as soon as there is a sufficient volume.

7.5.2 Dedicated Low-Flow (Minimal Drawdown) Technique

The pump and its tubing were installed previously and are dedicated to the well. Nitrogen gas is used to operate the pump. The flow rate is controlled by varying the gas pressure on the pump. The flow rate is set to less than or equal to the recharge rate of the well.

A minimum of two tubing water volumes (unless otherwise specified in the mini-SAP) need to be purged when using the Dedicated Low-Flow technique. Calculate tubing water volumes using the following formula.

one tubing water volume = $\pi r^2 h * 28310$ milliliter (mL)/ft^3

where:

$\pi = 3.14$
r = radius of the sample tubing (ft) = [diameter of sample tubing (in) x 1(ft)/12 (in)] / 2
h = tubing length (ft)

The following formulas are provided on the “Field Measurement Log For Groundwater Collection” to calculate the volume of water in one tubing length.

Tubing Diameter

1/4” outer diameter, 0.17” inner diameter: 4.5 mL/ft X ____ (length of tubing in ft) = ____ mL
3/8” outer diameter, 0.25” inner diameter: 9.6 mL/ft X ____ (length of tubing in ft) = ____ mL

Set the purge rate to less than or equal to the recharge rate of the well. A purge rate of < 1000 mL/minute (min) is desired. Monitor the water level periodically and adjust the purge rate to minimize drawdown in the well to less than 0.1 meters. It is especially important to minimize disturbance to the water in the well when measuring the water level. Record groundwater field parameters a minimum of four readings per tubing volume. Samples are collected after a minimum of two tubing water volumes are purged and the groundwater field parameters are stabile. See Section 7.6 for more information on collecting groundwater field parameter data. (Note: Wait a minimum of 24 hours before sampling with a newly installed dedicated low-flow pump.)

7.6 Measuring Groundwater Field Parameter Data

Connect the flow-through cell to the pump tubing. The flow-through cell houses the probes used to measure most of the groundwater field parameters (pH, SC, temperature, ORP, DO). Water pumped from the well flows through the cell and contacts the probes. Turbidity is measured with a portable turbidimeter.

Groundwater field parameters are identified in the min-SAP. All instruments and their corresponding probes are function checked prior to recording groundwater field parameters. Function check the instruments in accordance with FOP 05-02.
The samples are collected (see Section 7.8) after the well or tubing is properly purged and the groundwater field parameters are stable. The following stability criteria apply (if no others are listed in the mini-SAP):

Four consecutive measurements of:

- +/- 0.1 standard units for pH.
- +/- 1.0 degrees Celsius for temperature.
- +/- 5 percent for SC.
- +/- 10 percent or ≤ 5 nephelometric turbidity units for turbidity.

Additional field parameters may include ORP and DO, as required in the mini-SAP.
Field measurements are recorded on the Field Measurement Log for Groundwater Sample Collection (Attachment A).

7.7 Field Analysis

Samples may be collected for field analysis such as alkalinity, hexavalent chromium, nitrate, and ferrous iron. The field analyses will be specified in the mini-SAP. Digital titrators and colorimeters are used to perform the field analyses. Follow the manufacturer’s procedures when performing the analyses. Document the results on the Field Measurement Log for Groundwater Collection (Attachment A) or specific field analysis forms if applicable.

7.8 Sample Collection

Sample wells in the order specified in the mini-SAP. If an order is not specified, sample wells in the order from least contaminated to most contaminated. This will minimize the potential for cross-contaminating wells. If the potential for groundwater contamination has not been established at the site, sample wells from the up gradient direction toward the down gradient direction.

After the purging criteria have been met and groundwater field parameters are stable, collect the samples specified in the min-SAP. Collect samples in the order of decreasing volatilization sensitivity, as applicable:

Most Volatile:	1. Volatile organic compounds
	2. Total organic halogens
	3. Total organic carbon (TOC)
Least Volatile:	4. Base-neutral and acid extractable organic compounds
	5. Total metals
	6. Dissolved metals
	7. Phenols
	8. Cyanide
	9. Sulfate and chloride
	10. Turbidity (for laboratory analysis)
	11. Nitrate and ammonia
	12. Radionuclides
	13. Bacteria

7.8.1 Collection of Volatile Organic Analysis Fraction

Collect the VOC samples at a slow discharge rate to minimize volatilization. VOC sample containers should be filled at a rate not exceeding 100 mL/min. The sampling system may be modified in order to split the flow of water, such that the flow can be reduce to a rate of 100 ml/min or less.

Fill the sample vial completely. Pour the last few drops into the vial so that surface tension forms a convex meniscus on the water surface in the vial. Cap the bottle securely. Slight overflow will result in the elimination of air space in the bottle. Turn the bottle over and tap it gently to check for bubbles. If bubbles are present, repeat the procedure using a new sample container.

7.8.2 Filtration for Dissolved Metals and Major Cation Fractions

Sample fractions for dissolved metals and major cations (potassium, calcium, sodium, magnesium) require filtration to remove suspended solids. Use a disposable 0.45- micron in-line filter. Place over the discharge outlet. A minimum of over 500 mL should be drawn through the filter before sampling. Please note, filtration of samples is not limited to dissolved metals and cations. Consult the mini-SAP as to when filtration is required.

7.8.3 Equipment Blanks, Field Blanks, and Duplicate (Split) Samples

Collect any equipment blanks, field blanks, duplicate, or split samples as specified in the mini-SAP.

When collecting duplicate or split samples, it is important to take them in a consecutive order. That is, take all the VOC samples, then all the TOC samples, then all the metals, etc. Do not pull one complete set of samples and then pull another complete set. The groundwater chemistry may change during sampling and that could affect the consistency of the sample results.

7.8.4 Sample Handling and Documentation

Mark sample labels with the date and time of collection. Seal the sample containers with custody seal tape so they cannot be opened without breaking the tape. Place the sample containers inside sealable plastic bags. Immediately place samples requiring preservation into a sample cooler with ice or blue ice. Complete an ARCOC and transport the samples to SMO (see Section 7.11) when all field activities at the well location are completed.

7.9 Waste Disposal

Containerize, label, and process all wastes generated during purging, sampling, and decontamination activities in accordance with FOP 05-04.

7.10 Well Condition and Security

Remove all non-dedicated sampling equipment from the well and secure the equipment for transport. Place the casing cap on the well. Secure and lock the outer protective casing cap. Clean up the sampling site. Note on the Field Measurement Log for Groundwater Sample Collection (Attachment A) any problems with the well (such as missing or damaged hasps, locks, concrete pads, protective posts, obstruction inside the well casings, well access, etc.). Immediately inform the field support operations project leader of the problem.

7.11 Sample Shipment

SMO personnel verify with the field technician that the sample labels and the ARCOC are filled out correctly before they take custody of the samples from the field technician. SMO is responsible for shipping the samples to the designated analytical laboratory within the required holding times. For further information on sample handling refer to AOP 95-16.

7.12 Quality Assurance

FOP 05-02 is used as the quality assurance mechanism for operations described in this procedure.

8.0 RECORDS

A record of purging and sample collection activities is required. Forms that provide the required documentation are listed below:

- Field Measurement Log for Groundwater Sample Collection (Attachment A).
- Examples of Sample Label and Sample Identification Numbers (Attachment B).
- ARCOC.
- Groundwater Sample Collection Field Equipment Check Log (FOP 05-02, Attachment A).

The completed documentation is reviewed by the field support operations project leader. SMO will review sample labels and the ARCOC upon delivery of samples to the facility. All of the original documentation with the exception of the ARCOC and sample labels are, provided to the sampling coordinator for review before submittal to the project leader. The project leader reviews, approves, and returns the documentation to the sampling coordinator for transmittal to the Customer Funded Record Center.

9.0 REFERENCES

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ATTACHMENT A

FIELD MEASUREMENT LOG FOR GROUNDWATER SAMPLE COLLECTION

FIELD MEASUREMENT LOG FOR GROUNDWATER SAMPLE COLLECTION

Project Name:	Project No.:
Well I.D.:	Date:
Well Condition:	Weather Condition:

Method: Portable pump ____________________Dedicated pump _________________ Pump depth: _______

PURGE MEASUREMENTS

Depth to Water (ft)	Time 24 hr	Vol. (L/gal)	Temp (^oC)	SC (μS/cm)	ORP (mV)	pH	Turbidity (NTU)	DO (%)	Comments

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ATTACHMENT B

EXAMPLES OF SAMPLE LABEL AND SAMPLE IDENTIFICATION NUMBERS

EXAMPLES OF SAMPLE LABEL AND SAMPLE IDENTIFICATION NUMBERS

Sandia National Laboratories **Environmental Programs** Sample ID.# ___________________________ TYPE ____________ANALYSIS___________ LOCATION ____________________________ DATE ________________TIME ___________ PRESERVATIVES ______________________ COLLECTOR __________________________ SF2000-EPL(4-94)	SNL/NM 018562 SNL/NM 018563 SNL/NM 018564 SNL/NM 018565 SNL/NM 018566 SNL/NM 018567 SNL/NM 018568

**SANDIA NATIONAL LABORATORIES
LONG-TERM STEWARDSHIP**

FIELD OPERATING PROCEDURE

FOP 05-02

GROUNDWATER MONITORING EQUIPMENT FIELD CHECK FOR WATER QUALITY MEASUREMENTS

Author: Robert Ziock, Subject Matter Expert **Date:** 1/10/12

Approved: Michael Skelly, Project Leader **Date:** 10 Jan 12

Approved: Don Schofield, Field Support Operations Project Leader **Date:** 1/10/12

Approved: Pamela Puissant, Department Manager **Date:** 1/24/12

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] No*

EFFECTIVE DATE: 1/24/12

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Revision History

Revision	Review Date	Effective Date	Summary of Changes
1	9/24/2005	10/10/2005	New document
2	6/13/2007	8/16/2007	Formatting changes. Updated section 2, Roles and Responsibilities; section 3, Training Qualifications; section 9, References.
3	11/12/2009	11/12/2009	Formatting changes. Added work planning and control information to section 4.0, Health & Safety. Updated section 3, Training Qualifications; section 9, References.
4	1/10/2012	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 9, References.

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Table of Contents

TABLES

ATTACHMENTS

FORMS

EP 2009-ALW – Environmental Planning – Activity Level Work Evaluation Form
EP 2009-AUL – Environmental Planning – Authorized Users List
EP 2009-OJT – Environmental Planning – On-the-Job Training
EP 2009-WRA – Environmental Planning – Work Resumption Authorization Form

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Acronyms & Abbreviations

Ag	silver
AgCl	silver chloride
AOP	administrative operating procedure
°C	degrees Celsius
DI	deionized
DO	dissolved oxygen
Eh	oxidation-reduction
ES&H	Environmental, Safety & Health
FOP	field operating procedure
LTES	Long Term Environmental Stewardship
mg/L	milligrams per liter
µmhos/cm	micromhos/centimeter
mV	millivolt
NTU	nephelometric turbidity units
ORP	oxidation/reduction potential
pH	potential of hydrogen
PHS	primary hazard screening
PLA	plan
SAP	sampling and analysis plan
SC	specific conductivity
SMO	Sample Management Office
SNL/NM	Sandia National Laboratories, New Mexico
THA	task hazard analysis

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1.0 PURPOSE, SCOPE, AND OWNERSHIP

Purpose This field operating procedure (FOP) provides guidance on operating instruments used to measure groundwater field parameters during groundwater sampling of monitoring wells. Analytical parameters may include potential of hydrogen (pH), specific conductivity (SC), temperature, oxidation/reduction potential (ORP), turbidity, and dissolved oxygen (DO) content. The procedure also provides information on function checks and calibrations of instruments that act as the quality assurance mechanism for FOP 05-01, *Groundwater Monitoring Well Sampling and Field Analytical Measurements*.

Scope This procedure applies to groundwater field parameters measured during groundwater sampling activities conducted at Sandia National Laboratories, New Mexico (SNL/NM).

Ownership The Long-Term Stewardship Department is responsible for development, approval, and revision of this document.

2.0 ROLES AND RESPONSIBILITIES

The **project leader** is responsible for the following:

- Reviewing and recommending approval of this procedure.
- Providing overall coordination and management of the project.
- Providing a sampling and analysis plan (SAP) that meets prescribed regulatory or programmatic requirements.
- Reviewing completed field forms and data pertaining to the sampling activities.
- Reporting all information as may be 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. It details sampling activities for the field support operations project leader and field technicians. It summarizes sampling procedures, analytical parameters, field measured parameters, purge requirements, and waste management tasks. It also identifies monitoring well characteristics that may extend the sampling period (e.g., low yield wells, well construction issues, etc.).
- Coordinating sampling activities with the SNL/NM lead investigators, regulators, Kirtland Air Force Base and their environmental contractors, Sample Management Office (SMO), and field technicians.
- Providing the field support operations project leader with a copy of the mini-SAP.
- Coordinating sampling procedure variances with the project leader and the field support operations project leader.

- Reviewing completed field forms and data pertaining to the sampling activities.
- Ensuring that all data quality requirements are performed.
- Providing the project leader with a summary of field activities and sampling results.
- Reporting all information as may be required by regulations or directives.
- Submitting completed field forms to the Customer Funded Record Center and entry of relevant data to the Environmental Data Management System database.
- Managing, coordinating, and disposing of purge water and other waste generated from field operations in compliance with SNL/NM Corporate Policy ESH100 Environment Safety & Health, and FOP 05-04, *Groundwater Waste Management Plan.*
- Reviewing and providing recommendations for revisions to this procedure (if necessary).

The **field support operations project leader** is responsible for the following:

- Communicating with the sampling coordinator and the Field Support Operations Administrator regarding sampling activities.
- Supervising the field technicians.
- Reviewing training requirements for field technicians.
- Providing for on-the-job training of new field technicians.
- Assigning field technicians (qualified by training and experience) to conduct the activities described in this procedure.
- Coordinating sampling activities with the sampling coordinator, SMO, and field technicians.
- Ensuring the materials and equipment necessary to perform the work are available.
- Providing the field technicians with a copy of the mini-SAP.
- Reviewing field documentation.
- Maintaining the calibration records.
- Maintenance of the training matrix for all field personnel.
- Maintaining, reviewing, and revising all technical work documents.
- Notifying the project leader and sampling coordinator of unusual field conditions, wells requiring maintenance, or breach of well security.
- Reviewing and providing recommendations for revisions to this procedure (if necessary).

The **field technician** is responsible for the following:

- Completing all necessary and required training as specified by the field support operations project leader.
- Maintaining and decontaminating equipment.
- Field checking instruments according to the manufacturer's instructions and this procedure.
- Completing and reviewing field documentation forms.
- Providing recommendations for revisions to this procedure (if necessary).

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 Environmental Stewardship (LTES) Groundwater Monitoring Activities*.
- OJT for new field personnel performing field checks of water quality measurement equipment. Document training by completing On-the-Job Training form (EP 2009-OJT).
- Read and sign FOP 03-02, *LTES Groundwater Level Data Acquisition and Management*.
- Read and sign FOP 05-01, *Groundwater Monitoring Well Sampling and Field Analytical Measurements*.
- Read and sign FOP 05-03, *Groundwater Sampling Equipment Decontamination*.
- Read and sign FOP 05-04, *Groundwater Waste Management Plan*.
- Read and sign plan (PLA) PLA 05-09, *Groundwater Monitoring 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

Table 1. Training Course List (concluded)

Course Code	Course Title
PRS250	Advanced Pressure Safety
RAD230	Radiological Worker II Training
RAD230R	Radiological Worker II Retraining
RGH100	Crane, Rigging, Hoisting, & Hands-on Training

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 HEALTH AND SAFETY

A task hazard analysis (THA) has been performed on the activities described in this FOP as well as a hazard assessment survey performed by a SNL/NM industrial hygienist. They are detailed in the PLA 05-09, *Groundwater Monitoring Health & Safety Plan*. The THA was performed in conjunction with PHS SNL05A01241, *LTES Groundwater Monitoring Activities*.

An Activity Level Work Evaluation Form (EP 2009-ALW) was completed and approved by the department manager as required by administrative operating plan (AOP) AOP 09-10, *Work Planning and Control*.

A field technician or the field support operations project leader shall conduct a tailgate safety meeting and fill out a Tailgate Safety Meeting Form prior to the start of groundwater sampling activities as described in PLA 05-09, *Groundwater Monitoring Health & Safety Plan.*

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 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.

5.0 DEFINITIONS

dissolved oxygen (DO) content − The DO content of the water in milligrams per liter (mg/L) or percentage of oxygen saturation of the water at the indicated temperature.

specific conductivity (SC) − The ability of a cubic centimeter of water to conduct electricity. It varies directly with the amount of ionic substances in the water and is measured in micro-mhos per centimeter (µmhos/cm) at 25ºC. SC is an indirect measure of total dissolved solids in dilute solutions.

potential of hydrogen (pH) − The inverse log of the hydrogen ion concentration of a solution. A measure of the intensity for the basic or acidic condition of a solution. Range is from 0 to 14 with 1 being the most acidic. A neutral solution is at a pH of 7. Greater than 7 is considered basic.

oxidation/reduction potential (ORP) − The electric potential (usually measured in millivolts [mV]) required for the transfer of an electron from one molecule or element (the oxidant) to another molecule or element (the reductant). Used as a qualitative measure of the oxidation state of the molecules or compounds in solution. There are two types of ORP probes. A calomel probe and a silver (Ag)/silver chloride (AgCl) Ag/AgCl probe. Use the following equation to calculate oxidation-reduction (Eh) recorded with an ORP probe:

$$\text{Eh} = \text{ORP} + E^1$$

where:

E^1 (CALO MEL) = 244 mV

E^1 (Ag/AgCl) = 199 mV

temperature (°C) − The temperature of the water in degrees Celsius (°C).

turbidity (nephelo-metric) − The cloudiness in water due to suspended and colloidal organic and inorganic material. Water turbidity is measured in nephelometric turbidity units (NTUs).

6.0 EQUIPMENT AND MATERIALS

The following instruments are used to measure groundwater parameters:

- YSI Model 650 MDS with YSI 6920 Sonde and flow-through cell to measure °C, pH, SC, DO, and ORP.
- HACH 2100Q Turbidity Meter.

Equivalent instruments may be substituted. Follow manufacturer’s instructions for calibration and operation.

The following list includes field documentation forms, equipment and materials necessary to calibrate/check field analytical equipment, and/or to measure the parameters.

Documentation Forms:

- Groundwater Sample Collection Field Equipment Check Log (Attachment A).

Calibration Standard Solutions − Calibration standard solutions must be factory supplied and have expiration dates and/or lot number on the container.

- pH 4.00,7.00, and 10.00 solutions.
- electrical conductivity solutions: 500 to 1500 micromhos/centimeter (µmhos/cm) at 25°C reference solution.
- standard for oxidation/reduction potential: solution range 200 to 250 mV @ 25° C.
- Gelex® or equivalent turbidity standards: ≤10, 20, 100, and 800 NTU.

Additional Items:

- manufacturer's instructions/operator/manuals
- deionized water (DI)
- material safety data sheets

7.0 PROCEDURES

7.1 Equipment Calibration and Function Check

Review the mini-SAP to determine the groundwater field parameters to be measured and the corresponding instruments needed.

Calibrate or perform a function check on the instruments prior to measuring groundwater field parameters. Complete the Groundwater Sample Collection Field Equipment Check Log (Attachment A). Note the condition and quality of calibration standards. Make sure they have not exceeded their expiration date. Replace if necessary.

7.1.1 YSI 6920 Sonde and YSI 650 Multi Display System

The following is a set of instructions for calibrating a SI 6820 Sonde using a YSI 650 Multi Display System. The Sonde includes a DO, conductivity, pH, ORP and temperature probes. For additional information reference the manufacturer's manuals.

1) Turn unit on by pressing the green power button.
2) Use the Down arrow to "**Sonde menu**". Press enter (small arrow key, middle right).
3) Use large arrow keypad to select "**Calibrate**". Press enter.

4) To calibrate for DO:
    - Use large arrow keypad to select "**Dissolved Oxy**". Press enter.
    - Loosen calibration cup on Sonde.
    - Press enter after the barometric pressure is displayed.
    - Record both DO% and DO milligrams/liter (mg/L). Press enter.
    - Press "**Esc**" button.
5) To calibrate for conductivity:
    - Use large arrow keypad to select "**Conductivity**". Press enter.
    - Select "**SpCond**". Press enter.
    - Enter calibration value of standard solution according to the correct temperature (°C) on jar ("**1.278**" @ 20°C).
    - Place Sonde in calibration solution with calibration sleeve. Press enter.
    - Let the reading stabilize. Press enter.
    - Press "**Esc**" button.
    - Remove Sonde from calibration solution and rinse with DI water.
6) To calibrate for pH:
    - Use large arrow keypad to select "**ISE1 pH**". Press enter.
    - Use large arrow keypad to select "**3 point**". Press enter.
    - Place Sonde in pH 7 solution. Enter the value "**7.00**". Press enter.
    - Let the reading stabilize. Press enter.
    - Remove Sonde from pH 7 solution and rinse with DI water.
    - Place Sonde in pH 4 solution. Enter the value "**4.00**". Press enter.
    - Let the reading stabilize. Press enter.
    - Remove Sonde from ph 4 solution and rinse with DI water.
    - Place Sonde in pH 10 solution. Enter the value "**10.00**". Press enter.
    - Let the reading stabilize. Press enter.
    - Press "E**sc**" button.
    - Remove Sonde from pH 10 solution and rinse with DI water.
7) To calibrate for oxidation/reduction:
    - Use large arrow keypad to select "**ISE2 Orp**". Press enter.
    - Place Sonde in ORP calibration solution.
    - Enter the value from the ORP chart (Ag/AgCl column) according to the correct temperature (°C) ("**237.5**" @ 20°C). Press enter.
    - Let the reading stabilize. Press enter.
    - Press the "**Esc**" button twice.
8) Use large arrow keypad to select "**Sonde run**". Press enter. The instrument is now ready to measure groundwater field parameters.

No calibration is required for the temperature.

Reference the manufacturer's manual if an "**out of range message**" is displayed during the calibration process. It may be necessary to replace a probe that cannot be calibrated.

7.1.2 HACH 2100P Turbidimeter

The HACH 2100Q Turbidimeter should be calibrated every 3 months using StablCal Stabilized Formazin Standards followed by assigning values to Gelex® Secondary Turbidity Standards. Refer to the "Instrument and Procedure Manual" to perform this operation.

Once a value has been assigned to the Gelex® Secondary Turbidity Standards they can be used to perform a daily function check on the instrument prior to groundwater sampling activities. The following is a set of instructions to perform a function check prior to groundwater sampling activities:

1) Wipe the Gelex® Secondary Turbidity Standard cells (≤10, 20, 100, and 800 NTU) with a soft, lint-free cloth.
2) Apply a thin film of silicone oil and wipe with a soft cloth to obtain an even film over the entire surface of the cell.
3) Press: "**I/O**". The instrument will turn on. Place the instrument on a flat, sturdy surface.

4) Insert the Gelex® Secondary Turbidity Standard cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment. Close the lid.
5) Select manual or automatic range selection by pressing the "**RANGE**" key. The display will show "**AUTO RNG**" when the instrument is in automatic range selection.
6) Select signal averaging mode by pressing the "**SIGNAL AVERAGE**" key. The display will show "**SIG AV**" when the instrument is using signal averaging. Use signal average mode.
7) Press: "**READ**". The display will show "**----NTU**", then the turbidity in NTU. Record the value after the lamp symbol turns off.

If the reading is not within 5% of the previously established value, recalibrate the instrument with StablCal Stabilized Formazin Primary Standards as indicated in the "Instrument and Procedure Manual".

To obtain a turbidity value on a groundwater sample, fill an empty cell, wipe with a soft lint-free cloth, and repeat steps 2 through 7.

7.2 Equipment Maintenance

Store the equipment and perform routine maintenance as required by the manufacturer's instructions.

8.0 RECORDS

The completed documentation is reviewed by the field technician and sampling coordinator before it is provided to the project leader. The project leader reviews, approves, and transmits the documentation to the Customer Funded Records Center.

9.0 REFERENCES

HACH Company, CAT. NO. 46500-88, Portable Turbidimeter, Model 2100P, Instrument and Procedure Manual.

NovaLynx Corporation Model 230-355 Handheld Digital Barometer & Altimeter, Operating Instructions, File No. 355:440-1, (530) 823-7185 nova@novalynx.com.

Sandia National Laboratories, Corporate Policy ESH100 Environment Safety & Health, 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 05-03, *Groundwater Monitoring Equipment Decontamination* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, FOP 05-04, *Groundwater Monitoring Waste Management* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, PHS SNL05A01241, *Groundwater Monitoring Activities* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, PLA 05-09, *Groundwater Monitoring Health and Safety Plan* (latest edition), SNL/NM.

YSI Incorporated, YSI 650 Multiparameter Display System (MDS) Operation Manual

YSI Incorporated, YSI 6-Series (6920 V2 Sonde) Environmental Monitoring Systems

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ATTACHMENT A

GROUNDWATER SAMPLE COLLECTION FIELD EQUIPMENT CHECK LOG

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GROUNDWATER SAMPLE COLLECTION FIELD EQUIPMENT CHECK LOG Page 1 of 2

SNL/NM Project Name:	SNL/NM Project No.:
Calibrations done by:	Date:

Make & Model:

YSI 6820 Sonde (S/N) with DO, Ec, pH, ORP, and temperature probes: ____

YSI 650 MDS (S/N): ____

pH Calibration

pH Calibrated to (std):			pH sloped to (std):			
Reference value:	4.00		7.00		10.00	
	Value	Temp	Value	Temp	Value	Temp
1. Time:						
2. Time:						
3. Time:						
4. Time:						
Standard lot no.:						
Expiration date:						

SC Calibration

Reference Value:			Standard Lot No.:
	Value	Temp	Expiration Date:
1. Time:			
2. Time:			
3. Time:			
4. Time:			

ORP Calibration

Reference Value:			Standard Lot No.
	Value	Temp	Expiration Date:
1. Time:			
2. Time:			
3. Time:			
4. Time:			

DO Calibration

Calibration Value:	81% air saturation @ 5200 ft.	Atmospheric Pressure in Hg
1. Time:		
2. Time:		
3. Time:		
4. Time:		

GROUNDWATER SAMPLE COLLECTION FIELD EQUIPMENT CHECK LOG (continued) Page 2 of 2

SNL/NM Project Name:	Project No.:
Calibration done by:	Date:

TURBIDIMETER

Make & Model: HACH 2100P	Serial No. S/N

Reference Value	.1	20	100	800
Standard Lot No.				
1. Time				
2. Time				
3. Time				
4. Time				

Comments:

SANDIA NATIONAL LABORATORIES
LONG-TERM STEWARDSHIP

FIELD OPERATING PROCEDURE

FOP 05-03

GROUNDWATER MONITORING EQUIPMENT DECONTAMINATION

Author: Robert Ziock **Date:** 1/10/12
Robert Ziock, Subject Matter Expert

Approved: Michael F. Skelly **Date:** 10Jan12
Michael Skelly, Project Leader

Approved: Don Schofield **Date:** 1/10/12
Don Schofield, Field Support Operations Project Leader

Approved: Pamela Puissant **Date:** 1/24/12
Pamela Puissant, Department Manager

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*]	*No*

EFFECTIVE DATE: 1/24/12

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), department home page

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Revision History

Revision	Review Date	Effective Date	Summary of Changes
1	9/29/2005	10/10/2005	New document
2	6/13/2007	8/16/2007	Formatting changes. Updated section 2, Roles and Responsibilities; section 3, Training Qualifications; section 8, References.
3	11/12/2009	11/12/2009	Formatting changes. Added work planning and control information to section 4, Health and Safety. Updated section 3, Training Qualifications; section 8, References.
4	1/10/2012	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 8, References.

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TABLES

ATTACHMENTS

FORMS

EP 2009-ALW – Environmental Planning – Activity Level Work Evaluation Form
EP 2009-AUL – Environmental Planning – Authorized Users List
EP 2009-OJT – Environmental Planning – On-the-Job Training
EP 2009-WRA – Environmental Planning – Work Resumption Authorization Form

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Acronyms & Abbreviations

AOP	administrative operating plan
CHPD	closed head polyethylene drum
DI	deionized
ES&H	Environment, Safety and Health
FOP	field operating procedure
HNO_3	nitric acid
L	liter
LTES	Long Term Environmental Stewardship
ml	milliliter
OJT	on-the-job training
PHS	primary hazard screening
PLA	plan
PPE	personal protective equipment
SAP	sampling and analysis plan
SNL/NM	Sandia National Laboratories, New Mexico
THA	task hazard analysis

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1.0 PURPOSE, SCOPE AND OWNERSHIP

Purpose	This field operating procedure (FOP) provides instruction for decontaminating groundwater sampling equipment.
Scope	This procedure applies to all groundwater sampling equipment used during groundwater sampling activities conducted at Sandia National Laboratories/New Mexico (SNL/NM).
Ownership	The Long-Term Stewardship Department is responsible for development, approval, distribution, revision, and control of this document.

2.0 ROLES AND RESPONSIBILITIES

The **project leader** is responsible for the following:

- Reviewing and recommending approval of this procedure.
- Providing overall coordination and management of the project.
- Providing a sampling and analysis plan (SAP) that meets prescribed regulatory or programmatic requirements.
- Reviewing completed field forms and data pertaining to the sampling activities.
- Reporting all information as may be 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. It details sampling activities for the field support operations project leader and field technicians. It summarizes sampling procedures, analytical parameters, field measured parameters, purge requirements, and waste management tasks. It also identifies monitoring well characteristics that may extend the sampling period (e.g., low yield wells, well construction issues, etc.).
- Coordinating sampling activities with the SNL/NM lead investigators, regulators, Kirtland Air Force Base and their environmental contractors, Sample Management Office, and field technicians.
- Providing the field support operations project leader with a copy of the mini-SAP.
- Coordinating sampling procedure variances with the project leader and the field support operations project leader.
- Reviewing completed field forms and data pertaining to the sampling activities.
- Ensuring that all data quality requirements are performed.
- Providing the project leader with a summary of field activities and sampling results.
- Reporting all information as may be required by regulations or directives.
- Submitting completed field forms to the Customer Funded Record Center and entry of relevant data to the Environmental Data Management System database.

- Managing, coordinating, and disposing of purge water and other waste generated from field operations in compliance with SNL/NM Corporate Policy ESH100 Environment Safety & Health, and FOP 05-04, *Groundwater Waste Management Plan.*
- Reviewing and providing recommendations for revisions to this procedure (if necessary).

The **field support operations project 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 for the on-the-job training (OJT) of new field technicians.
- Assigning field technicians (qualified by training and experience) to conduct the activities described in this procedure.
- Ensuring the materials and equipment necessary to perform the work are available.
- Providing the field technicians with a copy of the mini-SAP.
- Reviewing field documentation.
- Maintenance of the training matrix for all field personnel.
- Maintaining, reviewing, and revising all technical work documents.
- Reviewing and providing recommendations for revisions to this procedure (if necessary).

The **field technicians** are responsible for the following:

- Completing all necessary and required training as specified by the field support operations project leader.
- Maintaining and decontaminating equipment.
- Completing and reviewing field documentation forms.
- Providing recommendations for revisions to this procedure (if necessary).

3.0 TRAINING QUALIFICATIONS

Personnel conducting equipment decontamination shall complete the following:

- Read applicable sections of SNL/NM Corporate Policy ESH100 Environment Safety & Health.
- Read primary hazard screening (PHS) SNL05A01241, *Long Term Environmental Stewardship (LTES) Groundwater Monitoring Activities*.
- OJT for new field personnel performing field checks of water quality measurement equipment. Document training by completing On-the-Job Training form (EP 2009-OJT).
- Read and sign FOP 03-02, *LTES Groundwater Level Data Acquisition and Management*.
- Read and sign FOP 05-01, *Groundwater Monitoring Well Sampling and Field Analytical Measurements*.
- Read and sign FOP 05-02, *Groundwater Monitoring Equipment Field Check for Water Quality Measurements.*
- 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 plan (PLA) PLA 05-09, *Groundwater Monitoring 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)
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MCH200	Hand and Power Tool Safety
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MED104	Heartsaver CPR
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PKX100	Basic Hazardous Material Transportation Training
PPE106	Personal Protective Equipment Training
PRS150	Pressure Safety Orientation
PRS250	Advanced Pressure Safety
RAD230	Radiological Worker II Training
RAD230R	Radiological Worker II Retraining
RGH100	Crane, Rigging, Hoisting, & Hands-on Training

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 HEALTH AND SAFETY

A task hazard analysis (THA) has been performed on the activities described in this FOP as well as a hazard assessment survey performed by a SNL/NM industrial hygienist. They are detailed

in the PLA 05-09, *Groundwater Monitoring Health & Safety Plan*. The THA was performed in conjunction with PHS SNL05A01241, *LTES Groundwater Monitoring Activities*.
An Activity Level Work Evaluation Form (EP 2009-ALW) was completed and approved by the department manager as required by administrative operating plan (AOP) AOP 09-10, *Work Planning and Control*.

A field technician or the field support operations project leader shall conduct a tailgate safety meeting and fill out a Tailgate Safety Meeting Form prior to the start of groundwater sampling activities as described in PLA 05-09, *Groundwater Monitoring Health & Safety Plan.*

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 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.

5.0 EQUIPMENT AND MATERIALS

The following list includes equipment and materials necessary for decontaminating groundwater sampling equipment:

- 55-gallon closed head polyethylene drum (CHPD)
- deionized (DI) water
- hand-held squirt bottles containing DI water
- non-phosphate laboratory detergent (e.g., Liqui-Nox®)
- hand-held squirt bottles containing non-phosphate laboratory detergent and tap water
- bottle brushes
- paper wipes
- plastic containers/buckets with lids
- personal protective equipment (PPE) − nitrile or latex gloves, chemical safety goggles, safety boots
- plastic garbage bags and waste storage containers
- Decontamination Log form (Attachment A)
- Groundwater Monitoring Waste Generation Log form (FOP 05-04, Attachment C)
- reagent grade nitric acid (HNO_3)

6.0 PROCEDURES

6.1 Equipment Decontamination

Wear the PPE specified in the PLA 05-09, *Groundwater Monitoring Health & Safety Plan*, when decontaminating equipment. The following is a list of groundwater monitoring equipment that needs to be decontaminated after each use and a set of instructions for performing the decontamination process:

- **static water level indicator**

  1) Wipe the last three feet of cable and probe with a paper wipe wetted with a 0.1% (1 milliliter/liter [1 ml/L]) Liqui-Nox®/DI or distilled water solution.
  2) Wipe the last three feet of cable and probe with a paper wipe wetted with deionized or distilled water.
  3) Wipe the last three feet of cable and probe with a clean, dry paper wipe.

- **portable non-dedicated piston pump system**
  Complete the following after the pump and its tubing have been removed from the well:

  1) Remove Sonde from the flow-through cell and rinse the probes (potential for hydrogen, specific conductance, dissolved oxygen, temperature, oxidation/reduction potential) with DI or distilled water.
  2) Seal the opening on the flow-through cell with the cap.
  3) Make sure all of the tubing used in purging and sampling remains connected to the pump and is included in the decontamination process (including the flow-through cell).
  4) Pump the following solutions through the tubing in the order listed below: Collect solution discharge in a 55-gallon CHPD.
     - ➢ 5 gallons of DI or distilled water mixed with 20 ml Liqui-Nox®.
     - ➢ 5 gallons of DI or distilled water.
     - ➢ 5 gallons of DI or distilled water mixed with 20 ml reagent grade HNO_3.
     - ➢ 3 times 5 gallons of DI or distilled water for a total of 15 gallons
  5) Rinse outside of pump tubing with DI or distilled water.
  6) Complete Decontamination Log form (Attachment A)
  7) Complete a Groundwater Monitoring Waste Generation Log form (FOP 05-04, Attachment C)

- **Dedicated Low-Flow Pump System**
  The pump and tubing are dedicated to the well and are not removed for decontamination. However, the tubing that is connected from the well to the flow-through cell is decontaminated. Complete steps 1 through 4 and steps 6 through 7 listed above.

- **Pressure Transducer and Cable**

  1) Wipe the cable (portion immersed in the water) and pressure transducer with a paper wipe wetted with a 0.1% (1ml/L) Liqui-Nox® /deionized or distilled water solution.
  2) Wipe the cable (portion immersed in the water) and pressure transducer with a paper wipe wetted with deionized or distilled water.
  3) Wipe the cable (portion immersed in the water) and pressure transducer with a clean, dry paper wipe.

6.2 Decontamination Waste

Handle all decontamination waste according to FOP 05-04, *Groundwater Waste Management Plan*.

7.0 RECORDS

The completed documentation is reviewed by the field support operations project leader and sampling coordinator before it is provided to the project leader. The project leader reviews and approves the documentation for transmittal to the Customer Funded Record Center by the sampling coordinator.

8.0 REFERENCES

Sandia National Laboratories, Corporate Policy ESH100 Environment Safety & Health, 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.

Sandia National Laboratories, Long-Term Stewardship, FOP 05-04, *Groundwater Monitoring Waste Management* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, FOP 09-05, *Conducting Slug Test Using Pressure Transducer and Data Logger* (latest edition), SNL/NM.

Sandia National Laboratories, Environmental Programs and Assurance Department, PHS SNL05A01241, *Long Term Environmental Stewardship Groundwater Monitoring Activities* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, PLA 05-09, *Groundwater Monitoring Health and Safety Plan* (latest edition), SNL/NM.

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Attachment A

Decontamination Log Form

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Portable Pump and Tubing / Water Level Indicator Decontamination Log Form

Project Name: ______________________	**Monitoring Well ID #** : ______________________________	**Date:** _______________

The following equipment was decontaminated at completion of sampling activities in accordance with FOP-05-03

Pump and Tubing Bundle ID #: _________________	**Water Level Indicator ID #:** _________________________
Personnel Performing Decontamination: ______________________________ ______ Print Name: Initial: ______________________________ ______ Print Name: Initial:	**Personnel Performing Decontamination:** ______________________________ ______ Print Name: Initial: ____________________ ______ Print Name: Initial:

Condition of Equipment

Pump: ___________________________ **Tubing Bundle**: ____________________________ **Water Level Indicator:** _________________________

List of Decontamination Materials

Distilled or **Deonized** (circle one) **Source:** ____ **Lot Number:** ____	**HNO_3** **Grade:** _________________________________ **UN #:** _________________________________ **Manufacturer:** _________________________________ **Lot Number:** _________________________________

SANDIA NATIONAL LABORATORIES
LONG-TERM STEWARDSHIP

FIELD OPERATING PROCEDURE

FOP 05-04

GROUNDWATER MONITORING WASTE MANAGEMENT

Author: Robert Ziock **Date:** 1/10/12
Robert Ziock, Subject Matter Expert

Approved: Joe Mauser **Date:** 1/10/12
Joe Mauser, Waste Water Discharge Project Leader

Approved: Michael F. Skelly **Date:** 10JAN12
Michael Skelly, Project Leader

Approved: Don Schofield **Date:** 1/10/12
Don Schofield, Field Support Operations Project Leader

Approved: Pamela Puissant **Date:** 1/24/12
Pamela Puissant, Department Manager

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*]	*No*

EFFECTIVE DATE: 1/24/12

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Revision History

Revision	Review Date	Effective Date	Summary of Changes
1	8/24/2005	10/10/2005	New document
2	6/13/2007	8/16/2007	Formatting changes. Updated section 3, Roles and Responsibilities; section 4, Training Qualifications; section 7, References.
3	11/12/2009	11/12/2009	Formatting changes. Added work planning and control information to section 5, Health and Safety. Updated section 4, Training Qualifications; section 7, References.
4	01/10/2012	01/24/2012	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 4, Training Qualifications; section 7, References.

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<u>EP 2009-AUL</u> – Environmental Planning – <u>Authorized Users List</u>
<u>EP 2009-OJT</u> – Environmental Planning – <u>On-the-Job Training</u>
<u>EP 2009-WRA</u> – Environmental Planning – <u>Work Resumption Authorization Form</u>
<u>SA2001-MWA</u> – <u>Less Than 90-Day Hazardous or Mixed Waste Accumulation Unit Inspection Form</u>

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Acronyms & Abbreviations

AOP	administrative operating plan
CFRC	Customer Funded Records Center
CHPD	closed-head poly drum
COC	chain of custody
decon	decontamination
FOP	field operating procedure
HWMF	Hazardous Waste Management Facility
OJT	on-the-job training
PHS	primary hazard screening
PLA	plan
PPE	personal protective equipment
RCRA	Resource Conservation and Recovery Act
RMWDR	radioactive or mixed waste disposal request
RMWMF	Radioactive and Mixed Waste Management Facility
SAP	sampling and analysis plan
SNL/NM	Sandia National Laboratories, New Mexico
THA	task hazard analysis
WDDR	waste description and disposal request

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1.0 PURPOSE, SCOPE, AND OWNERSHIP

Purpose This field operating procedure (FOP) provides instruction on managing waste generated during groundwater sampling activities. Waste is managed in compliance with Sandia National Laboratories, New Mexico (SNL/NM) Corporate Policy ESH100 Environment Safety & Health.

Scope The scope of this procedure is limited to management of well purge waste water, equipment decontamination (decon) waste water, Hach Accu-Vac ampule waste, personnel protective equipment (PPE) waste (nitrile or latex gloves), and paper wipes generated during groundwater monitoring activities at SNL/NM sites.

Ownership The Long-Term Stewardship Department is responsible for development, approval, distribution, revision, and control of this document.

2.0 BACKGROUND

The New Mexico Environmental Department has indicated that groundwater monitoring will be required to address the uncertainty of future impacts of contamination on groundwater. Specific wells within the Chemical Waste Landfill, Mixed Waste Landfill, Tijeras Arroyo Groundwater, Technical Area V, and the Burn Site Groundwater areas will be proposed for long-term monitoring. Separate requirements and site-specific monitoring plans will be developed and detailed upon regulatory approval of corrective measure evaluation, post-closure care provisions, long-term monitoring and maintenance plans, and other regulatory procedures.

Based upon historical results, groundwater sampling of monitoring wells is not expected to produce waste or contaminated materials requiring special handling or regulated disposal.

3.0 ROLES AND RESPONSIBILITIES

The **project leader** is responsible for the following:

- Reviewing and recommending approval of this procedure.
- Providing overall coordination and management of the project.
- Providing a sampling and analysis plan (SAP) that meets prescribed regulatory or programmatic requirements.
- Reviewing completed field forms and data pertaining to the waste management activities.
- Reporting all information as may be 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. It details sampling activities for the field support operations project leader and field technicians. It summarizes sampling procedures, analytical parameters, field measured parameters, purge requirements, and waste management tasks. It also identifies monitoring well characteristics that may extend the sampling period (e.g., low-yield wells, well construction issues, etc.).
- If needed, 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 the sampling activities.
- Ensuring that all data quality requirements are performed.
- Reviewing all analytical data used for waste characterization.
- Obtaining waste determination from the environmental 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 Waste Water Discharge project leader.
- Submitting disposal request to the appropriate department (Hazardous Waste Management Facility [HWMF], Radioactive and Mixed Waste Management Facility [RMWMF], Waste Water, and Solid Waste).
- Coordinating with the field support operations project leader for disposal and discharges (Work Request Form, Attachment A).
- 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 database.
- Reviewing and providing recommendations for revisions to this procedure (if necessary).

The **field support operations project leader** is responsible for the following:

- Communicating with the sampling coordinator regarding sampling activities.
- Supervising the field technicians.
- Verifying field technicians’ compliance with RCRA waste accumulation area training requirements.

- Providing for on-the-job training (OJT) of new field technicians.
- Assigning field technicians (qualified by training and experience) to conduct the activities described in this procedure.
- Coordinating sampling activities with the sampling coordinator, Sample Management Office, and field technicians.
- Ensuring the materials and equipment necessary to perform the work are available.
- Providing the field technicians with a copy of the mini-SAP.
- Reviewing field documentation.
- Maintenance of the training matrix for all field personnel.
- Maintaining, reviewing, and revising all technical work documents.
- Owner/manager/emergency coordinator of building 9925 RCRA Less Than 90-Day Waste Accumulation Area.
- Reviewing and providing recommendations for revisions to this procedure (if necessary).

The **field technicians** are responsible for:

- Completing all necessary and required training as specified by the field support operations project leader.
- Maintaining and decontaminating equipment.
- Managing and disposing of waste as directed by completed Work Request Forms and the field support operations project leader.
- Completing and reviewing field documentation forms.
- Providing recommendations for revisions to this procedure (if necessary).

The **waste water discharge project leader** is responsible for reviewing analytical data results of purge and decon water if requested by the sampling coordinator.

The **environmental protection representative** is responsible for reviewing analytical data results of purge and decon water if requested by the sampling coordinator.

The **health physicist** is responsible for reviewing analytical data results of purge and decon water if requested by the sampling coordinator.

4.0 TRAINING QUALIFICATIONS

Personnel conducting field activities shall complete the following:

- Read applicable sections of SNL/NM Corporate Policy ESH100 Environmental Safety & Health.
- Read primary hazard screening (PHS) SNL05A01241, *Groundwater Monitoring Activities*.
- OJT for new field personnel performing waste management activities. Document training by completing On-the-Job Training form (EP 2009-OJT).
- Read and sign FOP 03-02, *LTES Groundwater Level Data Acquisition and Management*.

- Read and sign FOP 05-01, *Groundwater Monitoring Well Sampling and Field Analytical Measurements*.
- 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 09-05, *Conducting Slug Test Using Data Logger & Pressure Transducer* (only necessary if conducting slug test).
- Read and sign Plan (PLA) PLA 05-09, *Groundwater Monitoring 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
RAD230	Radiological Worker II Training
RAD230R	Radiological Worker II Retraining
RGH100	Crane, Rigging, Hoisting, & Hands-on Training

CPR = Cardiopulmonary Resuscitation
ES&H = Environment, Safety and Health
HR = hour
OSHA = Occupational Safety and Health Administration
RCRA = Resource Conservation and Recovery Act

5.0 HEALTH AND SAFETY

A task hazard analysis (THA) has been performed on the activities described in this FOP as well as a hazard assessment survey performed by a SNL/NM industrial hygienist. They are detailed in the PLA 05-09, *Groundwater Monitoring Health & Safety Plan*. The THA was performed in conjunction with PHS SNL05A01241, *LTES Groundwater Monitoring Activities*.

An Activity Level Work Evaluation Form (EP 2009-ALW) was completed and approved by the department manager as required by administrative cooperating plan (AOP) AOP 09-10, *Work Planning and Control*.

A field technician or the field support operations project leader shall conduct a tailgate safety meeting and fill out a Tailgate Safety Meeting Form prior to the start of groundwater sampling activities as described in PLA 05-09, *Groundwater Monitoring Health & Safety Plan.*

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 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.

6.0 DESCRIPTION OF ACTIVITIES

The following is a description of activities for managing waste. They are summarized in a flowchart that is provided in Attachment B.

The project leader generates a SAP document. The SAP describes the groundwater sampling and waste management requirements identified in the permits or site closure plans for the specific sites.

The sampling coordinator prepares a mini-SAP that provides the field support operations project leader and field technicians with the information necessary for them to collect the groundwater samples and manage generated purge water, decon water, PPE, and paper wipe waste properly. The sampling coordinator may include additional sample analytical requirements that are not specified in the mini-SAP but are necessary to properly characterize the purge water, decon water, PPE, and paper wipe waste.

The field support operations project leader and field technicians use the information provided in the mini-SAP to perform the groundwater sampling at the designated locations. The waste generated during sampling activities includes purge water, decon water, PPE, paper wipes, and possibly HACH ampule waste. The mini-SAP identifies how the waste will be labeled and managed.

All purge water and decon water will be collected in separate 55-gallon closed-head poly drums (CHPDs). A label will be placed on each 55-gallon CHPD drum indicating the following:

- Initial Label Type (Hazardous or Non-regulated)
- Container Identification # (site-date-sequence)
- Project Name
- Waste Matrix (purge water or decon water)
- Accumulation Start Date
- Waste Owner, Organization, and phone number

All PPE and paper wipes will be placed in a plastic trash bag and waste receptacle. Both historical groundwater sample values and associated analytical results will be used to characterize the PPE and paper waste. A label will be placed on the waste container indicating the following:

- Initial Label Type (Hazardous or Non-regulated)
- Container Identification # (site-date-sequence)
- Project Name
- Waste Matrix (PPE, paper wipes)
- Accumulation Start Date
- Waste Owner, Organization, and phone number

The field technicians will complete a Groundwater Monitoring Waste Generation Log (Attachment C) as the waste is produced at each sampling location. The Groundwater Monitoring Waste Generation Log will identify:

- Container Identification # (site-date-sequence)
- Project Name
- Initial Label Type (Hazardous or Non-regulated)
- Waste Matrix (purge water, decon water, PPE, paper wipes)
- Container Type/Volume
- Volume of Waste
- Total Container Weight
- Chain of custody (COC) #: Sample#-Fraction
- Accumulation Start & Full Date
- Date Waste is moved to an Accumulation Area
- Accumulation Area Name

The 55-gallon drums will be moved to the appropriate storage area at completion of sampling activities. The 55-gallon drums labeled as containing hazardous waste will be placed in a RCRA Less Than 90-Day Waste Accumulation Area. The 55-gallon drums labeled as containing non-regulated waste will be kept at an area designated for non-regulated waste.

Because small quantities of PPE and paper wipe hazardous waste are generated, the 5-gallon poly bucket is temporarily stored in a Satellite Accumulation Area located in the groundwater sampling vehicle. It is moved to the appropriate waste accumulation area when the 5-gallon poly bucket is full.

HACH Accu-Vac ampule waste will be designated and labeled as a hazardous waste. It is collected in a 5-gallon poly bucket. Because small quantities of this waste are generated, it is temporarily stored in a Satellite Accumulation located in the groundwater sampling vehicle. It is moved to a RCRA Less Than 90-Day Waste Accumulation Area when the 5-gallon poly bucket is full.

The field support operations project leader will review and forward all completed field forms to the sampling coordinator at completion of sampling activities.

The field support operations project leader is responsible for managing and processing all hazardous and non-regulated waste once they are placed into the Building 9925 storage areas. Hazardous waste cannot exceed the 90-day hold time for a RCRA waste accumulation area. The sampling coordinator must complete a Less Than 90-Day Hazardous or Mixed Waste Accumulation Unit Inspection Form (SA2001-MWA) at time of the inspection. Completed inspection forms must be kept at or adjacent to the RCRA waste accumulation area. If an alternate 90-day area is used, the field support operations project leader will coordinate with the owner of the alternate site to ensure all regulatory and SNL/NM requirements are met.

To determine the disposal path of purge and decon water, the sampling coordinator reviews the analytical data results and determines whether:

1. The waste water discharge project leader (non-regulated waste) needs to review the data package.
2. The environmental protection representative (RCRA hazardous waste) needs to review the data package.
3. The health physicist (radioactive waste) needs to review the data package.

The disposal path of the PPE and paper wipe waste will also be determined by the disposal path of the purge and decon water analytical data results. The sampling coordinator prepares a data package that consists of a copy of the Waste Generation Log and a copy of the laboratory analytical results. The data package is then submitted to the appropriate person.

1. The waste water discharge project leader reviews the data package of non-regulated waste. After it is confirmed, the PPE and paper wipe waste can be disposed of as regular solid

waste. At this point there are three possible disposal paths for the non-regulated purge and decon water. They are:

- Discharge to the ground surface.
- Discharge to the sanitary sewer.
- Disposal through the HWMF.

Ground Surface Discharge — Only purge water may be discharged to the ground surface if the analytical data results meet 20.6.2 of the New Mexico Administrative Code. A surface discharge permit will be issued to the sampling coordinator by the waste water discharge project leader.

Sanitary Sewer Discharge — Purge and decon water that do not meet ground surface discharge requirements may be discharged to the sanitary sewer if the analytical data results meet the with Albuquerque Bernalillo County Water Utility Authority sanitary sewer discharge limits. A sanitary sewer discharge permit will be issued to the sampling coordinator by the waste water discharge project leader.

HWMF Disposal — Purge and/or decon water that does not meet sanitary sewer discharge requirements must be disposed through the HWMF. The waste water discharge project leader must provide a memo to the sampling coordinator stating that the purge and/or decon water must be disposed through the HWMF.

The sampling coordinator reviews the response given by the waste water discharge project leader and coordinates with the field support operations project leader for the authorized disposal of the purge and decon water accordingly.

If the purge and/or decon water must be disposed through the HWMF, then the sampling coordinator will fill out and submit a HWMF Waste Description and Disposal Request (WDDR) form. A copy of the laboratory analytical results (electronic or hard copy) must also be submitted to the HWMF.

If a permit was issued for discharge (ground surface or sanitary sewer), then the sampling coordinator will submit a Work Request Form (Attachment A) to the field support operations project leader to perform the discharge. The Work Request Form includes the following information:

- Project Name.
- Discharge Permit #.
- Indicate where the water will be discharged (ground surface or sanitary sewer).
- Number of containers to be emptied.
- Total volume of water to be discharged.
- Container identification numbers.
- Date of Discharge.

- Who completed the discharge.

The field support operations project leader receives the Work Request Form from the sampling coordinator to discharge the purge and/or decon water. The field support operations project leader schedules a field technician to discharge the purge and/or decon water as indicated in the Work Request Form. The completed Work Request Form is returned to the sampling coordinator by the field support operations project leader.

2. The environmental protection representative reviews the data package and verifies that the purge and decon water are a hazardous RCRA waste. The PPE and paper wipes associated with the purge and decon water will also be treated as hazardous waste. The sampling coordinator makes arrangements to dispose of the purge water, decon water, PPE, and paper wipes through the HWMF. All HACH Accu-Vac ampule wastes, regardless of sample location, will be treated as hazardous waste.

   To dispose of the waste, the sampling coordinator must fill out a HWMF WDDR form. The form is accessible on-line at:

   http://www-irn.sandia.gov/esh/hwmf_prgrm/wddr.htm

   The HWMF may request a data package on the waste in addition to the WDDR form.

3. The health physicist reviews the data package and verifies that the purge and decon water are a radioactive waste. The PPE and paper wipes associated with the purge and decon water will also be treated as radioactive waste.

   To dispose of the waste, the sampling coordinator must fill out a RMWMF Radioactive or Mixed Waste Disposal Request (RMWDR) form. The form is accessible on-line at:

   https://info.sandia.gov/esh/esh_center/Radioactive_Waste/RegWaste_dforms.htm

   The RMWMF may request a data package on the waste in addition to the RMWDR form.

7.0 REFERENCES

Sandia National Laboratories, Corporate Policy ESH100 Environment Safety & Health, 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.

Sandia National Laboratories, Long-Term Stewardship, FOP 09-05, *Conducting Slug Test Using Pressure Transducer and Data Logger* (latest edition), SNL/NM.

Sandia National Laboratories, Environmental Programs and Assurance Department, PHS SNL05A01241, *Long Term Environmental Stewardship Groundwater Monitoring Activities* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, PLA 05-09, *Groundwater Monitoring Health and Safety Plan* (latest edition), SNL/NM.

Attachment A

Work Request Form

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Work Request Form

Sampling Coordinator : ______________________ **Date Issued:**________________			
Project Name:			
Discharge Permit #:			
Discharge to: (Ground Surface or Sanitary Sewer)			
Number of containers to be emptied:			
Total Volume of Waste to be discharge:			
Container I.D. #'s:			
Date of Discharge:			
Discharge Completed by: (Field Technician)	Print Name: ____________________ Signature: ________________________	Print Name: ____________________ Signature: ________________________	Print Name: ____________________ Signature: ________________________

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Attachment B

Groundwater Waste Management Flowchart

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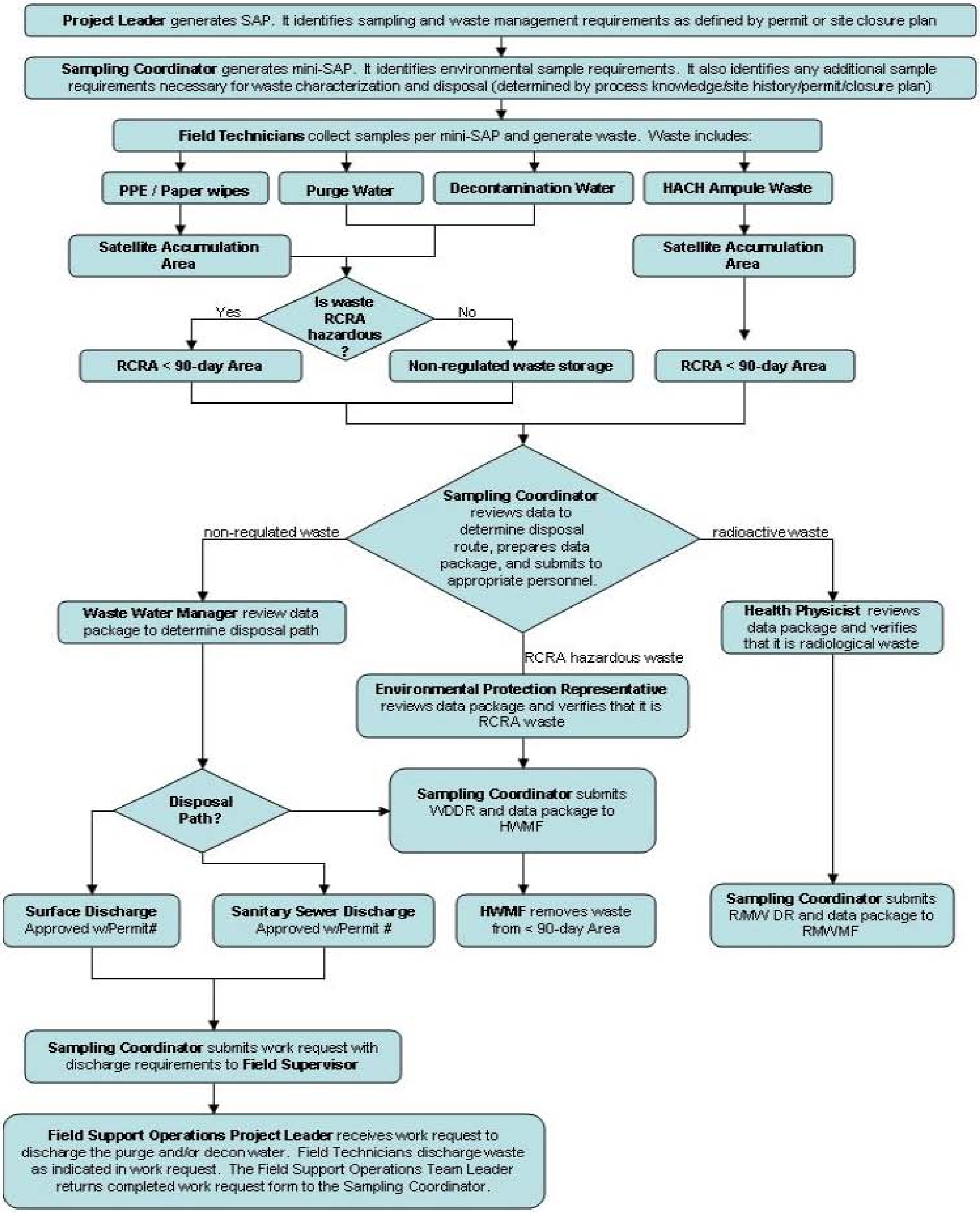
Groundwater Waste Management Flowchart



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Attachment C

Groundwater Monitoring Waste Generation Log

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Groundwater Monitoring Waste Generation Log

Waste Generator : ________________ **Phone:** _______________ project leader: ________________

Project Name			
Container ID # (site-date-sequence)			
Initial Label Type (Hazardous or Non-Regulated)			
Waste Matrix (purge water, decon water, HACH Accu-Vac ampule)			
Container Type / Volume			
Volume of Waste			
Total Container Weight			
COC#: Sample#-Fraction	______ ______ ______ ______ ______ ______ ______	______ ______ ______ ______ ______ ______ ______	______ ______ ______ ______ ______ ______ ______
Accumulation Date	Start: Full:	Start: Full:	Start: Full:
Date Waste Moved to Accumulation Area			
Accumulation Area Name			
Comments:			

SANDIA NATIONAL LABORATORIES
LONG TERM STEWARDSHIP DEPARTMENT (4142)

SAMPLE MANAGEMENT OFFICE (SMO) SAMPLE HANDLING, PACKAGING AND SHIPPING

LABORATORY OPERATING PROCEDURE

LOP 94-03

Revision 6

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Prepared By: 11.05.2013
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SMO Project Coordinator, 4142

Reviewed By: Don W Watenpaugh 11/5/13
Don Watenpaugh — Date
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Packaging and Shipping Project Leader, 10269

Reviewed By: 11/5/2013
Patrick Beall — Date
Radiation Protection Radiological Engineer, 4128

Approved By: Pam Puissant 11/12/13
Pam Puissant, — Date
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: 11/12/13

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
0	01/14/1994	New Document
1	05/14/1996	Administrative Updates
2	09/17/1998	Administrative Updates
3	12/16/2003	Organization ownership change from Sandia ES&H to Environmental Restoration Project
4	6/26/2007	Changed revision cycle from 2 to 3 years. Organization ownership change from Sandia Environmental Restoration Project to Sandia ES&H Organization.
5	6/29/2011	Programmatic revisions include the addition of the Sample Management Analysis Request Tool (SMART) and the addition of Industrial Hygiene (IH) sampling. Other revisions are definition updates, sentence structure, grammar, and formatting. Additions include Revision History page, tracking box and footnote disclaimer.
6	11/12/2013	Programmatic revisions include improvements to ARCOC processing, the addition of Bioassay sampling and changes to Industrial Hygiene (IH) sampling to include the use of the Radiological Process Knowledge Form (SF 6951-RRF). Chem101, PKX050, and OJT for HWMF Operations coordination were added to training requirements. A buddy system has been mandated for Friday work schedules. The chemicals were removed from use in addition to removing the use of the fume hood. The two Emergency Evacuation routes have been described with additional evacuation information. A requirement to stage first time samples waiting RCT survey in the RMA has been added. Other revisions include updating language to reflect current program elements and requirements.

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ATTACHMENTS

AUTHORIZED USERS LIST

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ACRONYMS AND ABBREVIATIONS

AOP	Administrative Operating Procedure
ARCOC	Analysis Request/Chain-of-Custody Record
COC	Chain-of-custody
DOE	U.S. Department of Energy
DOT	U.S. Department of Transportation
EPA	U.S. Environmental Protection Agency
IATA	International Air Transport Association
IH	Industrial Hygiene
LOP	laboratory operating procedure
MOU	memorandum of understanding
OP	operating procedure
PHS	Primary Hazard Screening
RCT	Radiation Control Technician
RMA	Radioactive Management Area
RPDP	Radiation Protection Dosimetry Program
RPPM	Radiation Protection Procedures Manual
RPSD	Radiation Protection Sample Diagnostics
SARF	Sample Analysis Request Form
SMART	Sample Management Analytical Request Tool
SMO	Sample Management Office
SMO-QAPP	Sample Management Office Quality Assurance Project Plan
SNL/NM	Sandia National Laboratories/New Mexico
SOW	Statement of Work

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1.0 PURPOSE, SCOPE, AND OWNERSHIP

The main purpose for this Laboratory Operating Procedure (LOP) is the following:

- Sample integrity is maintained during the packaging and shipping processes by Sandia National Laboratories/New Mexico (SNL/NM)/ Sample Management Office (SMO).
- SMO packaging processes meet regulatory requirements with applicable state, federal, local and international transportation regulations, U.S. Department of Energy (DOE) regulations and Sandia Environment, Safety and Health (ES&H) policies and procedures.

1.2 Scope

This document applies to all SMO employees and contractors responsible for the packaging and/or shipping of samples via a common carrier at SNL/NM.

1.3 Ownership

The SMO is responsible for the development, approval, distribution, revision and control of this document. Suggestions for improvement to this document should be submitted to the SMO.

2.0 RESPONSIBLE INDIVIDUALS AND ORGANIZATIONS

The following individuals and organizations have the specified responsibilities with regard to samples submitted to the SMO Packaging Facility for packaging and shipping.

Requesting Organization(s): Organizations that require SMO services are responsible for communicating requirements to the SMO at least two weeks prior to sample collection. This allows sufficient time to notify the analytical laboratories and provide sample kits (Bottle Orders) for sampling events. SMO customers, including field personnel, are responsible for providing complete sample documentation, completed Analytical Request Chain-of-custody (ARCOC) or Sample Request Analysis (SARF) forms (Attachment A, B and C), and Radiological Survey Forms (Attachment F) as applicable. In addition, SMO customers must notify the SMO of any special shipping requirements (i.e., late delivery, short hold times, limited quantity, etc.).

Packaging Coordinators: Trained SMO Packaging Coordinators (refer to Training section) are responsible for the packaging of all samples shipped to contract analytical laboratories through the Receiving/Mail & Material Movement Organization (10261) commonly referred to as "Shipping and Receiving". SMO Packaging Coordinators ensure that samples are properly processed, stored, and packaged for shipment to analytical laboratories in accordance with the current *SMO Quality Assurance Project Plan* (SMO-QAPP), DOE, US Department of Transportation (DOT), and International Air Transportation Administration (IATA) requirements. In addition, the Packaging Coordinators shall ensure that sample custody is maintained and documented in accordance with the current SMO-QAPP and the *Sample Management and Custody Administrative Operating Procedure* (AOP-95-16).

Hazardous Material Packaging Consultants: The Logistics Risk Management consultants in the Sandia National Laboratories Shipping and Receiving organization are responsible for oversight and guidance and are the final authority in the shipment of all samples processed at the SMO Packaging Facility as described in Section 7.3 of this document.

Radiological Control Technicians (RCTs): Radiation Protection Program RCTs are responsible for surveying all non-exempt samples prior to shipping. See Section 7.1 for a discussion regarding sample exemption.

3.0 TRAINING QUALIFICATIONS

Requesting Organization: Customers requesting SMO services on a regular basis shall read this procedure and sign the SMO Sample Handling, Packaging and Shipping LOP Authorized Users List. Customers who use SMO services occasionally are not required to sign the Authorized Users List.

Packaging Coordinator: Required training is listed in the Primary Hazard Screen (PHS) document (PHS 972834764), *SMO Packaging Facility Operations* (current revision). Upon completion of required training, the SMO Packaging Coordinator is qualified to process and package samples for shipping through Logistics Risk Management SNL Shipping and Receiving/Mail & Material Movement organizations. This training is in compliance with the Code of Federal Regulations 49 CFR Subpart H, Part 172.700-704. The Packaging Coordinator must complete training listed in Table 1.

Table 1 - Training Course List

Course Code	Course Title
CHM100	Chemical Safety
CHM 101	Chemical Safety Training Update for GHS
CHM103	Site Specific Chemical Safety
ESH 100	ES&H Awareness
ESH 200	Safety Management
ENV102	OSHA Health & Safety Basic Training – Occasional Worker (24 HR)
ENV103	OSHA Health & Safety Training Refresher (8 HR)

Table 1 - Training Course List (concluded)

Course Code	Course Title
ENV112	Hazardous Waste & Environmental Management Training
PKX050	Site Specific Packaging and Transportation of Hazardous Materials Training
PKX100	Basic Hazardous Material Transportation Training
PKX112	Basic Hazardous Waste Transportation Training
PKX115	Basic Hazardous Material Driver Training
PKX130	Basic International Air Transportation (IATA) Training
PKX111	Basic Radioactive Materials Transportation Training
PKX211	Advanced Radiological Materials Transportation Training
PPE106	Personal Protective Equipment Training
RAD102	General Employee Radiological Training (May be replaced with RAD210, Rad230 or SNL qualified RCT training)
RAD210	Radiological Worker I Training
OJT	HWMF Operations Coordination (Contingency Plan and MOU)

4.0 HEALTH AND SAFETY

Hazards Identification:
A complete listing of hazards is identified in PHS 972864764, *SMO Packaging Facility Operations* (current version). In addition, Sampling/Field Technicians shall inform SMO Packaging Coordinators and Packaging Facility Staff of other known potential hazards associated with samples to be packaged and shipped by SMO. Hazards associated with sample handling and packaging activities include, but are not limited to, the following:

- Chemical hazards
- Radiological hazards
- Physical hazards
- Biological hazards (etiologic agents) are a remote possibility not incident to normal working conditions.

5.0 EQUIPMENT AND MATERIALS

The Packaging Facility equipment and materials include but are not limited to the following list.

Equipment

- Eberline E600 Survey Meter with attached SHP380AB (or equivalent) is used by the RCTs in accordance with the Radiation Protection Operating Procedure RPO-03-31 for indication only. Daily instrument source checks are performed in accordance with the

Radiation Protection Procedures Manual (RPPM), Chapter 12, Attachment 12-1.

- Work Table Commercial Scale with a capacity of 150 lbs/68 kg. for weighing shipping containers/coolers.
- Various laboratory refrigerators and freezers for sample storage, including storage of containers/coolers, and Blue Ice^{TM}.

Materials

- Re-sealable plastic bags and bubble bags (assorted sizes)
- Insulated plastic ice chests/coolers (preferably without a spout)
- Absorbent packing material (i.e., Powersorb™ Universal Sorbent Pads)
- Large plastic bags
- Strapping tape
- Strapping bands
- Crimping supplies
- Custody seal tape
- Labels: Up arrows, “Fragile” and various hazard and handling DOT/IATA approved labels (i.e., “Flammable”, “Corrosive”, etc.)
- Cooling material (frozen) (i.e., Blue Ice^{TM}). In the event that cooling material is not readily available, ice may be substituted, provided that the ice is double-bagged, sealed and placed in the cooler with the bag opening facing up.
- Gloves: chemical resistant gloves and disposable, latex, polyvinyl chloride (PVC), nitrile, etc.
- Safety glasses
- Lab coat or coveralls
- Steel toed shoes
- Thermoluminescent dosimeter (TLD) for personnel involved in handling radioactive materials and samples which have not been screened by an RCT.

6.0 SMO PACKAGING FACILITY (BUILDING 928) HOURS OF OPERATION

SMO Packaging Coordinators are available at the Packaging Facility (Building 928) on ***Monday through Thursday from 7AM to 5PM*** **and on** ***Friday, from 7AM to 12***. The SMO customer shall make special arrangements with the SMO in advance for sample delivery to Building 928 during non-standard hours of operation. The SMO uses a buddy system on Fridays due to historical light work load and single person packaging support. The SMO Packaging Coordinator working on Friday, shall notify one other SMO support staff when work is complete and the coordinator is leaving for the day/week.

If same day packaging and shipping is required, samples shall be delivered to the SMO Packaging Facility, Building 928, ***before 11:00AM***. This allows sufficient time for the SMO Packaging Coordinator to complete the packaging process and meet corporate shipping deadlines. If corporate shipping deadlines cannot be met and sample integrity or customer requirements may be compromised, the SMO Packaging Coordinator or authorized representative may deliver samples to the FedEx drop-off located at the Albuquerque International Sunport (3720 Spirit Dr SE). The SMO Packaging Coordinator or authorized representative will deliver the entire consignment to the carrier for transport. **This applies to non-hazardous samples only.** Non- hazardous samples are described in the RPPM, Chapter 6: "*ES&H samples are exempted from the requirements of the RPPM if it is reasonably expected that the exterior of the sample container exhibits contamination levels less than those listed in Attachment 6-1, 'Radioactive Contamination Limits', and if dose rate on contact with the container is less than 0.5 mrem per hour"*.

7.0 SAMPLE RECEIPT AND PACKAGING PROCEDURES

If samples are determined to be hazardous (flammable, corrosive, radioactive, etc.), the type of hazard shall be documented and SMO shall be notified prior to receiving samples. The SMO Packaging Coordinator shall follow the procedures outlined in Section 7.2 of this procedure.

7.1 Receiving Non-Hazardous Samples

- A minimum of one chain-of-custody must accompany submitted samples. The SMO Packaging Coordinator or SMO Support Staff shall be present during the acceptance of samples at the SMO Packaging Facility in Building 928 (Section 7.1.1).

- If the samples are not packaged for shipment immediately and refrigeration is required, the SMO Packaging Coordinator shall refrigerate the samples to maintain the temperature at $\leq 6^{o}$C until shipment to the analytical laboratory.

- Items containing Radioactive Material shall be stored in the area posted "Controlled Area" and "Radioactive Material Area (RMA)".

- Based on process knowledge, IH media and samples are shipped as non-regulated.

- An RCT shall survey all non-exempt samples before they are shipped. Samples that are exempt and *do not* require an RCT survey prior to shipment include non-hazardous ground water samples, IH and any other samples with a Clearance – Radiological Process Knowledge Form (SF 6951-RRF) on file and exempt bioassay samples. Exempt bioassay samples that meet the following definitions and other criteria are qualified for the exemption

(Diagnostic Specimen Exemptions: under IATA DGR 2005, Amendment III):

- Specimens are those collected directly from humans or animals, including, but not limited to, excreta, secreta, blood and its components, tissue and tissue fluid, swabs, and body parts being transported for purposes such as research, diagnosis, investigational activities, disease treatment, and prevention.
- A patient or animal specimen is considered exempt if there is a minimal likelihood that pathogens are present. In determining whether a patient or animal specimen has a minimal likelihood that pathogens are present, an element of professional judgment is required to determine if a substance is exempt. This judgment should be based on known history, symptoms, and individual circumstances of the source, human or animal, and endemic local conditions.
- Examples of specimens which may be transported under the exemption include the blood or urine tests to monitor cholesterol levels, blood glucose levels, hormone levels, PSA tests, tests required to monitor organ function such as heart, liver, or kidney function for humans or animals with non-infectious diseases, or therapeutic drug monitoring; tests conducted for insurance or employment purposes, biopsies to detect cancer; and antibody detection in humans or animals.

- The SMO Packaging Facility shall not ship any samples that have specific activity above that specified by the Nuclear Regulatory Commission (NRC) license of the contract analytical laboratory and/or IATA Section 6. Or, those samples that fail to meet the definition and other criteria under IATA DGR 2005 for Diagnostic Specimen Exemptions.

7.1.1 SAMPLE CHECK- IN

7.1.1.1 Documentation

Ensure that all applicable documentation is correct and present. Nitrile gloves shall be worn when handling all samples.

- Ensure that the information on the Analysis Request and Chain-of-custody (ARCOC) and/or SARF for the IH program, Radiation Protection Dosimetry Program (RPDP) or the Onsite Laboratory (RPSD) forms are legible. (Attachment A, B, C and D). Illegible request forms will not be accepted.

- Ensure that sample containers are labeled and that all labels are legible and complete. The container labels in Attachment E are required for customers submitting samples on the ARCOC. Illegible sample labels will not be accepted.

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- If samples are from a radiological controlled area, the Sample Team Member shall have the *Radiological Survey* documentation (Attachment F) and/or the radiological survey number present upon sample delivery to the SMO Packaging Facility.

- If samples are destined for the RPSD laboratory for analysis (i.e., gamma spec, alpha, beta, etc.), a completed *Onsite Laboratory (RPSD) SARF* from the Sample Team Member shall accompany the samples to the laboratory. (Attachment D).

- The Sample Team Member shall work with the SMO Packaging Coordinator or other SMO support staff to ensure that all sample containers and request forms are correct and sample information is cross-referenced and complete.

7.1.1.2 Sample Container Inspection

The SMO Packaging Coordinator and/or the Support Staff have the authority to refuse samples that do not meet the following criteria.

- Ensure that containers are clean, sealed and intact.
- Verify that each sample container has double containment in a re-sealable plastic bag. Glass containers shall be placed in sealed plastic bubble bags.
- Verify that no sample containers are leaking or broken.
- Verify that all sample container lids are secured with SNL approved custody tape. 40 ml glass vial containers for aqueous samples like groundwater, and IH containers, are exempt from this requirement. The container(s) are placed in a plastic bag and the bag, not the container(s), is secured with the custody tape.
- Verify that all sample containers are labeled with a complete and legible label. The label in Attachment E is required for all samples submitted on an ARCOC.
- Bioassay sample packaging must consist of the following three components:
  - a leak-proof primary receptacle(s);
  - a leak-proof secondary packaging, and
  - outer packaging of adequate strength for its capacity, mass and intended use, and with at least one surface having minimum dimensions of 100mm x 100mm.

7.1.1.3 Chain-of-custody Verification

Contract Laboratory SMO ARCOC

Review the Contract Laboratory ARCOC to verify that:

- The controlled document ARCOC form is used. This is SMO 2012-ARCOC (4-2012).
- The *ARCOC Number* is present.
- The *Page Numbers* are present and correct.
- The *Project Information* is correct (Dept. No./Mail Stop, Project/Task Manager, Service Order Number). This information can be found in the Project Management table in the Sample Management Analysis Request Tool (SMART) application. This information is uploaded from the Electronic Bottle Order and from the Electronic ARCOC.
- The *Analytical Lab* information is correct (Lab Contact, Lab Destination). External analytical lab information is uploaded from the Electronic Bottle Order and from the Electronic ARCOC. (Note that Analytical Lab information is not uploaded for the IH and RPDP SARF COC.) The SMO Packaging personnel may verify this information by checking the Lab Management Table in the SMART Application.
- The analytical laboratory *Contract Number* is correct. Contract numbers are found in the Lab Management Table in the SMART Application and are uploaded from the Electronic Bottle Order and the Electronic ARCOC.
- The *Project/Task No. and Customer group* are correct. This information is found in the SMART Application's most recent Project Management Tables and is uploaded from the Electronic Bottle Order and the Electronic ARCOC.
- The appropriate box in the Radiation Material Area, *RMA,*section is marked. If "Yes" is checked in the *RMA* box, verify that the Rad Survey data is provided and write "*Rad Survey Data Provided*" under *Comments*.
- The appropriate *Sample Disposal* box has been marked.
- The appropriate *Turnaround Time* (TAT) has been indicated. 30 days is a normal TAT. For a *Negotiated TAT*, the Sample Team Member shall indicate the number of days requested for the rush delivery (i.e., 3, 5 etc.). The 3, 7-Day TAT and Negotiated TAT prior authorization is completed when the Electronic Bottle Order is submitted.
- The *Special Instructions/QC Requirements* section of the ARCOC may be used by the Sample Team Member to indicate special instructions for SMO and/or the analytical laboratory.
- The *Sample Team Members* section has been completed. A signature for each field technician listed is required.
- The sample information on the ARCOC matches the information on the sample labels. All information must be cross-referenced and correct. If there are multiple sample containers assigned to a *Sample No.-Fraction*, verify that the number of sample containers submitted matches those indicated in the *Container-Volume* column (i.e., 3X40 mL will have three 40 mL containers for that sample).
- To relinquish samples go to Section 7.1.1.4.

IH SARF Chain-of-custody

Review the IH SARF Chain-of-custody Report (Attachment B) to verify:

- The *IH Survey ID* is present.
- The *Sample Location* is indicated.
- The *Lab receiving sample* is indicated.
- The *Submitted By and Submission Date* are indicated and correct.
- The *Charge code* (Project/Task number) is correct. This information is found in the most recent Project Management Table of the SMART Application.
- The *Send Report To* and *Phone* are indicated.
- The *Attention of* line is completed with the name of the person that will receive the final data package. The *Email* line is filled in with this person's e-mail address. Filling in the *Fax* line is optional.
- The *Analysis requested* is completed.
- The *General Comments to lab personnel* are included. Generally, contact information and more specific sample information are indicated in this section.
- The *IH Sample #, Col. Date, Turn-Around-Time* and *Matrix* are indicated.
- The *Sample Comments* lists an SMO Sample Number-Fraction to correspond to each *IH Sample #*.
- The sample information on the SARF concurs with the samples delivered. All information must be cross-referenced and correct.
- Note that the IH SARF COC is 2 pages. The second page is the signature page for received and relinquished signatures. Verify that the IH Survey ID number is listed on the second page.
- To relinquish samples go to Section 7.1.1.4.

RPDP SARF

Review the RPDP SARF COC to verify that:

- The *RPDP Batch No* is present.
- The *Page Numbers* are *present and* correct.
- The *Customer Name, Organization (4121), Phone (845-DOSE/284.5598), email (dosimetry@sandia.gov) and program Name (Radiation Protection Dosimetry Program* are listed.
- The *Analytical Lab* information is correct (Lab Contact, Lab Destination). The analytical laboratory *Contract Number* is correct. Contract numbers are found in the Lab Management Table in the SMART Application.

- The appropriate *Turnaround Time* (TAT) has been indicated. Normal (N) TAT is 30 days, Rush (R) TAT is 15 days and Urgent (U) TAT is 7 days.
- *Date Needed By* has been filled.
- The *Special Instructions/QC Requirements* section is completed. This section should include “Biological Sample – Treat with Caution” and an RPDP contact (name and phone).
- The sample information on the RPDP SARF matches the information on the sample labels. All information must be cross-referenced and correct. If there are multiple sample containers assigned to a *Customer Sample ID*, verify that the number of sample containers submitted matches those indicated in the *Qty/Tot Volume Tare Wt* column.
- Assure that the Electronic Data Delivery, *EDD*, box is checked either yes or no.
- To relinquish samples go to Section 7.1.1.4.

7.1.1.4 Relinquishing Samples

Samples are relinquished after the chain-of-custody document is deemed correct and complete. A Chain-of-custody (on ARCOC) or Submitted by customer (on IH SARF) or person delivering the bioassay samples shall sign and date the appropriate *Relinquished By* box.

- The SMO representative shall sign and date the appropriate *Received By* box. For the ARCOC and RPDP SARF, this box is located directly below the *Relinquished by* signature. For the IH SARF, this box is located directly across from the *Relinquished by* signature.
- For the ARCOC, authorized SMO personnel shall sign the *SMO Authorization* line.
- The IH SARF is 2 pages. The second page is the signature page for relinquishing and receiving samples. Verify that the IH Survey ID number is indicated on the second page.
- Assure that all pages of the chain-of-custody are identified with the ARCOC Number, IH Survey ID Number or RPDP Batch Number.
- Give a copy of the ARCOC, IH SARF or RPDP SARF to the customer.
- Enter all pertinent ARCOC, IH SARF or RPDP SARF information in the Sample Management Log (Attachment G).
- Place the original ARCOC, IH SARF or RPDP SARF and accompanying documentation with the samples in preparation for packaging and shipping

7.1.2 SAMPLE PACKAGING FOR ENVIRONMENTAL OR IH SAMPLES

Follow the listed steps when packaging Environmental or IH samples in preparation for shipment. Nitrile gloves shall be worn when handing all samples.

1. Verify that:
   - Each sample container is in a re-sealable plastic bag and that the bag is sealed.
   - Glass containers are placed in a re-sealable bubble bag and the bag is sealed. Glass containers larger than the plastic bags are surrounded with cushioning material (bubble wrap) or placed in a "bubble" sleeve.

2. Place absorbent material (Powersorb™ Universal Sorbent Pads) in the bottom of the cooler and place an appropriately sized waterproof plastic bag on top of the absorbent. If the cooler contains water samples, place an additional sorbent pillow inside the plastic bag at the bottom of the cooler. Place the protected sample container(s) in the large plastic bag and alternate glass containers with plastic containers or padding. This step does not pertain to IH samples which are packaged and shipped in boxes.

3. Place frozen cooling material (Blue Ice™) in the cooler for samples requiring temperature preservation. Avoid direct contact of the Blue Ice™ with sample containers (Blue Ice™ is often below 0^oC). Insulate 40 mL vials with other sample containers or with water filled plastic bottles if necessary. (40 mL vials freeze or break if they are not protected due to the small volume of liquid.)

4. Pack the container with cushioning material to minimize the possibility of breakage from dropping or severe shock. Seal the large plastic bag containing samples with duct or plastic tape or a strong rubber band.

5. Log on to Web Shipper and follow the on-line instructions to complete the Web Shipper form.
   - A Shipper Number is automatically assigned. All coolers or boxes shipped to the same laboratory may be included on one shipper. Each laboratory location requires a separate shipper. Write the Shipper Number on the associated chain(s) of custody.
   - Assign a cooler or box number (i.e., #1, #2, #3, etc.) for each cooler that is being shipped to the same location.
   - For the Description on the Line Item List enter the cooler/box number, volume of the cooler and the corresponding ARCOC number, IH Survey ID or RPDP Batch Number of the samples packed in the cooler. (Example, "Cooler #1; 3.0 Cu. Ft.; COC606877")
   - Do not close the Web Shipper form at this time. Go to Step 6. The Web Shipper will be completed after Steps 6-11 are completed in Step 12.
   - Write the Shipper Number on all chain(s) of custody associated with the shipper.

6. Make a copy of the original ARCOC or SARF. Place the original ARCOC or SARF in a re-sealable plastic bag and place it on top of the large sealed and closed plastic bag

containing the samples. Close the cooler/box lid.

7. If the cooler has a drain plug, secure the drain in the closed position with packing or duct tape.

8. Secure the cooler lid with packing tape in two locations. Use strapping bands completely around the cooler in two locations.

9. Mark and label the cooler as indicated below:
    - Place the appropriate analytical laboratory pre-printed address label on the cooler lid and ensure that the ARCOC number, IH Survey ID number or RPDP Batch Number on the shipper form matches the ARCOC number or IH Survey ID number on the address label. Cover with transparent tape.
    - Place "Up" arrow labels on two opposing sides of all coolers containing liquids. Put a "Fragile" label on the cooler lid for all coolers with glass containers.
    - Record the shipper number and cooler number (i.e., "1 of 3") on the packing tape located on the cooler lid. Do not write directly on the cooler lid. The packing tape will be removed and the cooler will be re-used.

10. Weigh each cooler and record the weight in the Sample Management Log (Attachment G). The weight of the cooler will also be recorded in the *Comments* section of the Web Shipper (Step #12).

11. Measure the dimensions of the cooler and record the total cubic feet on the appropriate line on the address label. The cooler dimensions will also be recorded in the *Comments* section of the Web Shipper (Step #12).

12. Return to the Web Shipper form.
    - In the *Comments* section, enter the weight of the filled cooler (Step #10), the volume and dimensions of the cooler (Step #11) and enter: "FED-EX FIRST OVERNIGHT". (Example, "44LBS; 3.0 CU. FT.=23X14X15; DO NOT RE-PACK, FED-EX FIRST OVERNIGHT")
    - The completed shipper is approved electronically and is automatically sent to Shipping and Receiving.
    - Print 1 copy of the completed shipper. This copy is taken to Shipping and Receiving with the corresponding cooler(s). After samples are shipped, Shipping and Receiving will send a notification with a completed shipper that includes a tracking number.

13. Transport the cooler(s) and or boxes to Shipping and Receiving, Building 957 and present the Web Shipper form to the Service Clerk. DOT driver training is not required to transport non-hazardous samples but is required for the transportation of hazardous and

radioactive samples.

14. Complete all entries in the SMO Packaging Facility Sample Management Log (Attachment G).

15. Documentation submitted to the SMO data administrator may be submitted by one of the following options:

- Make a pink and yellow copy of the ARCOC or the IH SARF COC. Place these copies, a copy of the shipper and other documentation pertaining to the ARCOC or IH SARF COC in a large Ziploc™ bag and deliver to the SMO data administrator. The yellow copy is part of the laboratory data package and is sent to the SNL Records Center. The pink copy is kept at SMO as a reference copy.
- Or, the completed chain(s) of custody with associated documentation pertaining to the ARCOC or IH SARF COC is scanned and placed in the SMO COCsPackaging folder on the SMO shared drive.

7.1.3 SAMPLE PACKAGING FOR BIOASSAY SAMPLES

1. RPDP will deliver unsealed, packed bioassay coolers for shipment. The bioassay samples will be packed per Procedure No: RPDP-03-03 (current issue). The samples will be accompanied with an RPDP SARF.
2. Inspect the sample containers to verify that:
    - Sample containers are in a re-sealable plastic bag and that the bag is sealed.
    - Verify that no sample containers are leaking.
    - Verify that sample containers are labeled with a complete and legible label.
3. Follow Steps 1-15 in Section 7.1.2 (Sample Packaging for Environmental or IH Samples)

7.2 Receiving Hazardous Samples

The following steps must be followed when hazardous samples are received. Refer to *Shipment Planning* at Ship an Item (Web Shipper).

- If a sample is determined to be hazardous upon field-testing, via process knowledge and/or during sample survey, SMO personnel shall be notified of the specific hazard prior to sample delivery.
    - If unanticipated radioactive material or removable surface contamination exceeding RPPM Attachment 6-1 limits is determined refer to the **EMERGENCY PROCEDURES** section of this document.

- All samples that are labeled radioactive by an RCT shall be stored in the Radioactive

Material Area (a labeled refrigerator).

  - General area radiation levels are maintained at a level that does not require posting as a controlled area (<100 mrem per year or about ≤0.05 mrem/hr). If total samples present increase the general area dose rate to greater than 0.05 mrem/hr, the Controlled Area boundaries will be extended to reduce radiation exposure to personnel. The SMO support RCT is responsible for all radiation related postings or meter readings.

- Radiation screening shall be conducted on a representative sample fraction at the onsite RPSD laboratory to determine the total isotopic activity.
  - A sample that has a specific activity of 0.002 µCi/g or less may be packaged and shipped as an environmental sample with a disclaimer (Attachment H).
  - A sample that has a specific activity greater than 0.002 µCi/g shall be regarded as a radioactive material [IATA Section 6.0.1.3(a)].

- If a sample is determined to be radioactive, it must be within the allowable limits of the contract laboratory NRC license. Copies of NRC licenses documenting allowable radioactive limits are on file at the SMO Packaging Facility or electronically from SMO personnel or on the SMO shared drive in the NRC Licenses folder. The SMO Packaging Facility shall not accept a radioactive sample for packaging if it does not meet the allowable limits of the contract laboratory NRC license. The SMO Packaging Coordinator shall notify the customer when a sample is refused and the customer shall take custody of the sample immediately upon notification.

- If a sample is determined to be radioactive and within the allowable limits of the contract laboratory license, the SMO Packaging Coordinator and/or Packaging Facility staff will prepare the sample for shipment in accordance with IATA Section 10 as it applies to the packaging, marking, labeling, certification and documentation requirements for Class 7 radioactive materials (Preparing Hazardous Samples section). The SMO Packaging Coordinator shall deliver these radioactive samples SNL Shipping and Receiving for packaging and shipping.

- Radioactive samples shall only be delivered to SNL Shipping and Receiving by SMO staff in accordance with Corporate Procedure SCM100.3.19, *Movement of Hazardous Material.*

7.3 Preparing Hazardous Samples

The SMO Packaging Coordinator or SMO Packaging Staff shall take samples deemed hazardous, but not classified as radioactive materials, to Sandia Shipping and Receiving for packaging and shipping. The SMO Packaging Coordinator shall reference the section, *"How to*

Use the Regulations" in the current edition of the IATA Dangerous Goods Manual when preparing hazardous samples for delivery to Shipping and Receiving (Building 957). The steps below provide general guidance when preparing hazardous samples for delivery to Shipping and Receiving.

1. Inform the Hazardous Material Packaging Consultant of the specific hazards and the composition and amount of the specific hazards (Hazardous Material Packaging, Packaging Engineers).

2. Prepare and complete the required forms:
   - Web Shipper
   - Information for Hazardous Material Shipments at *Shipment Planning*
   - Print two copies of the completed *Web Shipper* and *Hazardous Material Shipments* form. (One copy is for Shipping and Receiving and one is for SMO records.)
   - Attach the completed *Hazardous Material Shipments* form to the *Web Shipper.*

3. Complete the remainder of the *SMO Use Only* section on the ARCOC form (Attachment A) with the required information.

4. Deliver the following to Shipping and Receiving, Building 957:
   - Hazardous samples
   - Completed *Web Shipper* and attached *Hazardous Material Shipments* form

5. The Hazardous Material Packaging Consultant shall review the *Web Shipper* and attached *Hazardous Material Shipments* form for completeness and correctness. If errors are found, the package shall be refused for shipment until all corrections are made.

EMERGENCY PROCEDURES

An emergency is defined as an unplanned, significant event or condition that requires time-urgent actions from emergency response resources to ensure the:

- Health and safety of Members of the Workforce and the public.
- Protection of the environment.
- Security of operations.

Emergency procedures shall be in accordance with established SNL policies and procedures. Refer to ESH100.3.1, *Prepare for and Manage Emergencies*, and refer to the current version of the *Contingency Plan for the Hazardous Waste Management Facility* (HWMF) (PLA94-23). The SMO Packaging Facility is a close neighbor to the HWMF, directly east. When Tone Alert

Radio (TAR) alerts go off at the HWMF, they also go off in Building 928. SMO personnel in the SMO Packaging Facility shall follow the procedure outlined in the HWMF Contingency Plan when TAR alerts sound.

General post-incident actions by involved personnel are:

- Stop activity.
- Secure the scene to prevent further injury or damage, if it is safe to do so.
- Evacuate if necessary. The SMO facility has two evacuation routes, one to the north and one to the south. Personnel shall check the wind socks at the HWMF and make a determination on the most appropriate evacuation route going away from potential inhalation hazards.
- Do not disturb the scene.
- Notify Department Manager.

Actions required when discovering unanticipated radioactive material or removable surface contamination are listed below.

- Discovery of unanticipated radioactive material will require employees to place work and materials into a safe configuration and exit the building pending a radiological survey and evaluation by SNL Radiation Protection and Facility Management. To mitigate the chance of unanticipated radioactive material, all new samples without process knowledge, are placed in the facility RMA prior to RCT scans.

- Discovery of material with removable surface contamination in excess of RPPM Attachment 6-1 limits for removable surface contamination will require employees to place work and materials into a safe configuration and exit the building pending a radiological survey and evaluation by SNL Radiation Protection and Facility Management.

The following information from ESH100.3.1, *Prepare for and Manage Emergencies* provides guidance for both emergency and non-emergency situations.

Table 8. Hazardous Materials Release, Emergency

1. At SNL/NM, call 911 or 844-0911 (cellular).
2. Evacuate and isolate the immediate area. Keep personnel from walking or driving through the affected area. For an outdoor release, stay upwind to avoid fumes.
3. If it is possible to do so safely, determine the chemical and quantity spilled; provide this information to emergency responders.
4. Consult the Material Safety Data Sheets and area ES&H Safety Operating Procedures for information about the released material and provide to emergency responders.

Table 9. Hazardous Materials Release, Non-Emergency

1. For a non-emergency situation, call 311 or 844-6515 or 844-0311 (cellular). To clean up large spills, call the Non-Emergency Hotline (844-6515) for spill response and cleanup.
2. Clean up small spills generated by personnel, as long as you have the correct training and equipment. If you do not feel comfortable doing so, request advice or assistance from the Non-Emergency Hotline (844-6515).
3. Handle all spilled material, absorbents, neutralizers and contaminated PPE as chemical waste. Follow the requirements for containment, labeling, storage, and disposal request specified in ESH100.2.ENV.22, Manage Hazardous Waste at SNL/NM.

Call for Help:

- 911 or 844-0911 (cellular)
- Call 311 or 844-6515 or 844-0311 (cellular), Non-Emergency Phone Numbers, when observing or experiencing an unusual condition that does not appear to constitute an emergency.
- Call the appropriate phone number in (as soon as it is safe to do so) when:
    - An emergency condition is observed or experienced.
    - You are unsure whether you have an emergency or not.

8.0 REFERENCES

International Air Transport Association, Dangerous Good regulations, 52nd edition (updated annually), International Air Transport Association, Montreal, Canada.

Sandia National Laboratories Environment Safety and Heath, Corporate Processes and Procedures, https://my.sandia.gov/authsec/portal/cps/environmentalSafetyHealth

Sandia National Laboratories Quality Assurance Project Plan for the SNL/NM Sample Management Office, SMO QAPP, Current Revision, Sandia National Laboratories/New Mexico Sample Management Office, Albuquerque, New Mexico.

Sandia National Laboratories Sample Management and Custody Administrative Operating Procedure (AOP-95-16), Current Revision, Sandia National Laboratories/New Mexico, Albuquerque, New Mexico.

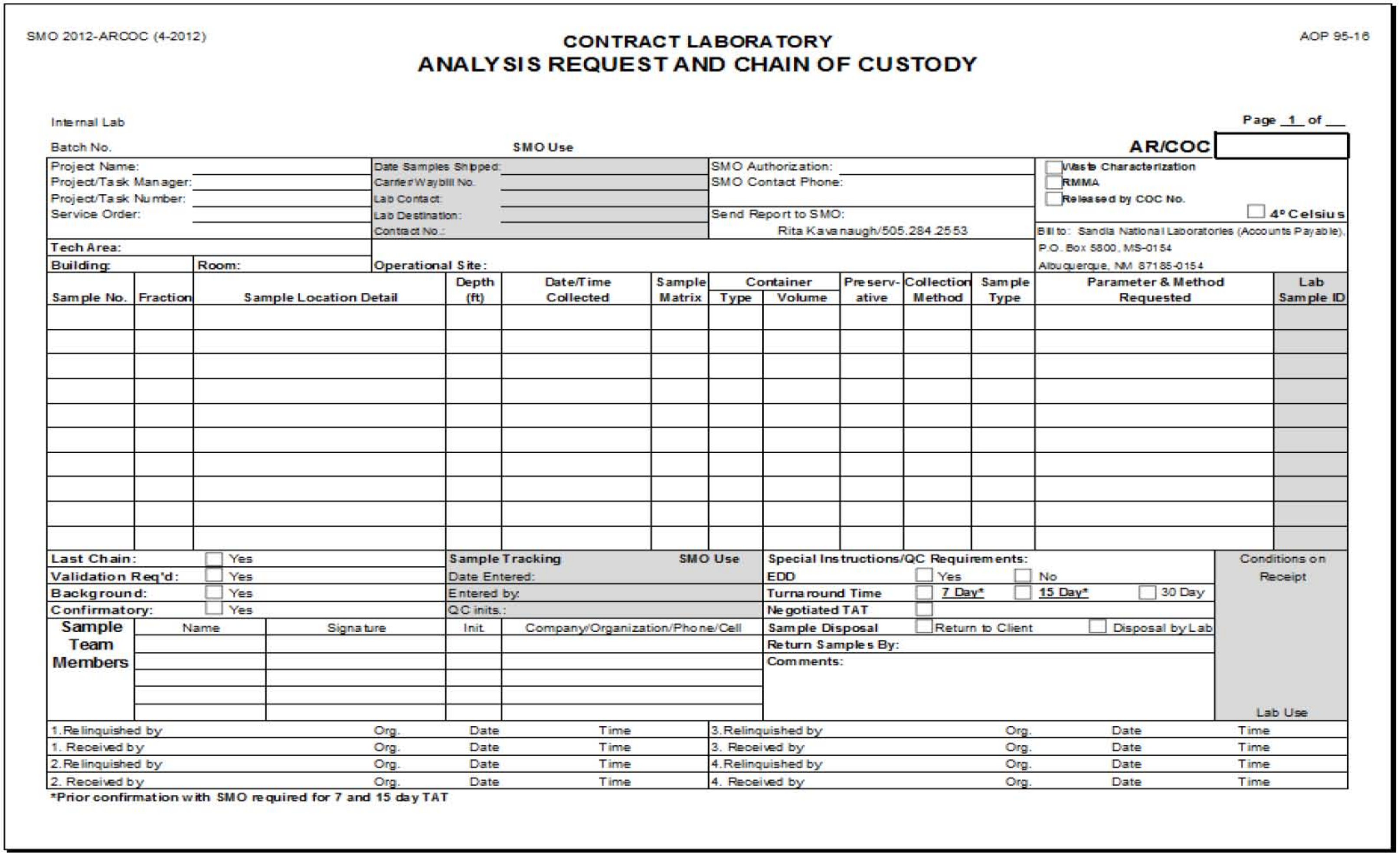
Sandia National Laboratories Statement of Work for Analytical Laboratories, Current Revision, Sample Management Office, Sandia National Laboratories/New Mexico (SNL/NM), Albuquerque, New Mexico.

Sandia National Laboratories Supply Chain Management Corporate Processes and Procedures, SCM100.3.6, https://my.sandia.gov/authsec/portal/cps/supplyChainManagement

US Environmental Protection Agency (EPA), Code of Federal regulations, CFR Title 40, (http://www.epa.gov/epahome/cfr40.htm).

ATTACHMENTS

ATTACHMENT A:
CONTRACT LABORATORY ANALYSIS REQUEST AND CHAIN-OF-CUSTODY
SMO 2012-ARCOC



SMO 2012-ARCOC (4-2012)

AOP 95-16

CONTRACT LABORATORY
ANALYSIS REQUEST AND CHAIN OF CUSTODY

Internal Lab

Batch No.

SMO Use

Page _1_ of ___

AR/COC

Project Name:
Project/Task Manager:
Project/Task Number:
Service Order:

Date Samples Shipped:
Carrier/Waybill No.
Lab Contact:
Lab Destination:
Contract No.:

SMO Authorization:
SMO Contact Phone:

Send Report to SMO:
Rita Kavanaugh/505.284.2553

☐ Waste Characterization
☐ RMMA
☐ Released by COC No.
☐ 4° Celsius

Bill to: Sandia National Laboratories (Accounts Payable),
P.O. Box 5800, MS-0154
Albuquerque, NM 87185-0154

Tech Area:

Building: **Room:**

Operational Site:

Sample No.	Fraction	Sample Location Detail	Depth (ft)	Date/Time Collected	Sample Matrix	Container Type	Container Volume	Preserv-ative	Collection Method	Sample Type	Parameter & Method Requested	Lab Sample ID

Last Chain: ☐ Yes
Validation Req'd: ☐ Yes
Background: ☐ Yes
Confirmatory: ☐ Yes

Sample Tracking **SMO Use**
Date Entered:
Entered by:
QC inits.:

Special Instructions/QC Requirements:
EDD ☐ Yes ☐ No
Turnaround Time ☐ 7 Day* ☐ 15 Day* ☐ 30 Day
Negotiated TAT ☐
Sample Disposal ☐ Return to Client ☐ Disposal by Lab
Return Samples By:
Comments:

Conditions on Receipt

Lab Use

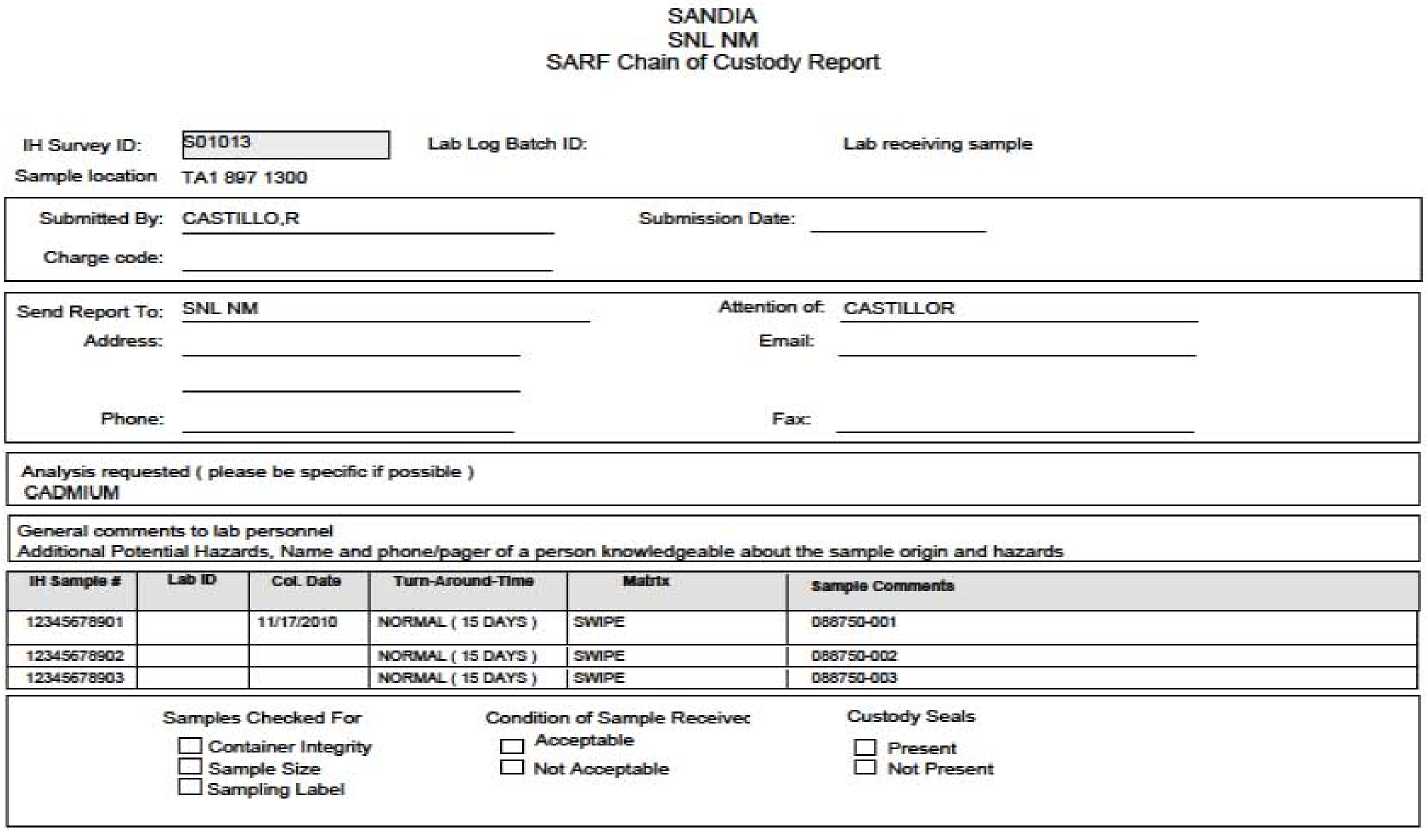
Sample Team Members	Name	Signature	Init.	Company/Organization/Phone/Cell

1. Relinquished by	Org.	Date	Time	3. Relinquished by	Org.	Date	Time
1. Received by	Org.	Date	Time	3. Received by	Org.	Date	Time
2. Relinquished by	Org.	Date	Time	4. Relinquished by	Org.	Date	Time
2. Received by	Org.	Date	Time	4. Received by	Org.	Date	Time

***Prior confirmation with SMO required for 7 and 15 day TAT**

This ARCOC is for example purposes only. The ARCOC currently in effect is posted on the 4100 Controlled Documents homepage (SMO 2012-ARCOC).

ATTACHMENT B: Industrial Hygiene (IH) SARF Chain-of-custody



SANDIA
SNL NM
SARF Chain of Custody Report

IH Survey ID: S01013
Lab Log Batch ID:
Lab receiving sample
Sample location TA1 897 1300

Submitted By: CASTILLO,R
Submission Date:
Charge code:

Send Report To: SNL NM
Attention of: CASTILLOR
Address:
Email:
Phone:
Fax:

Analysis requested (please be specific if possible)
CADMIUM

General comments to lab personnel
Additional Potential Hazards, Name and phone/pager of a person knowledgeable about the sample origin and hazards

IH Sample #	Lab ID	Col. Date	Turn-Around-Time	Matrix	Sample Comments
12345678901		11/17/2010	NORMAL (15 DAYS)	SWIPE	088750-001
12345678902			NORMAL (15 DAYS)	SWIPE	088750-002
12345678903			NORMAL (15 DAYS)	SWIPE	088750-003

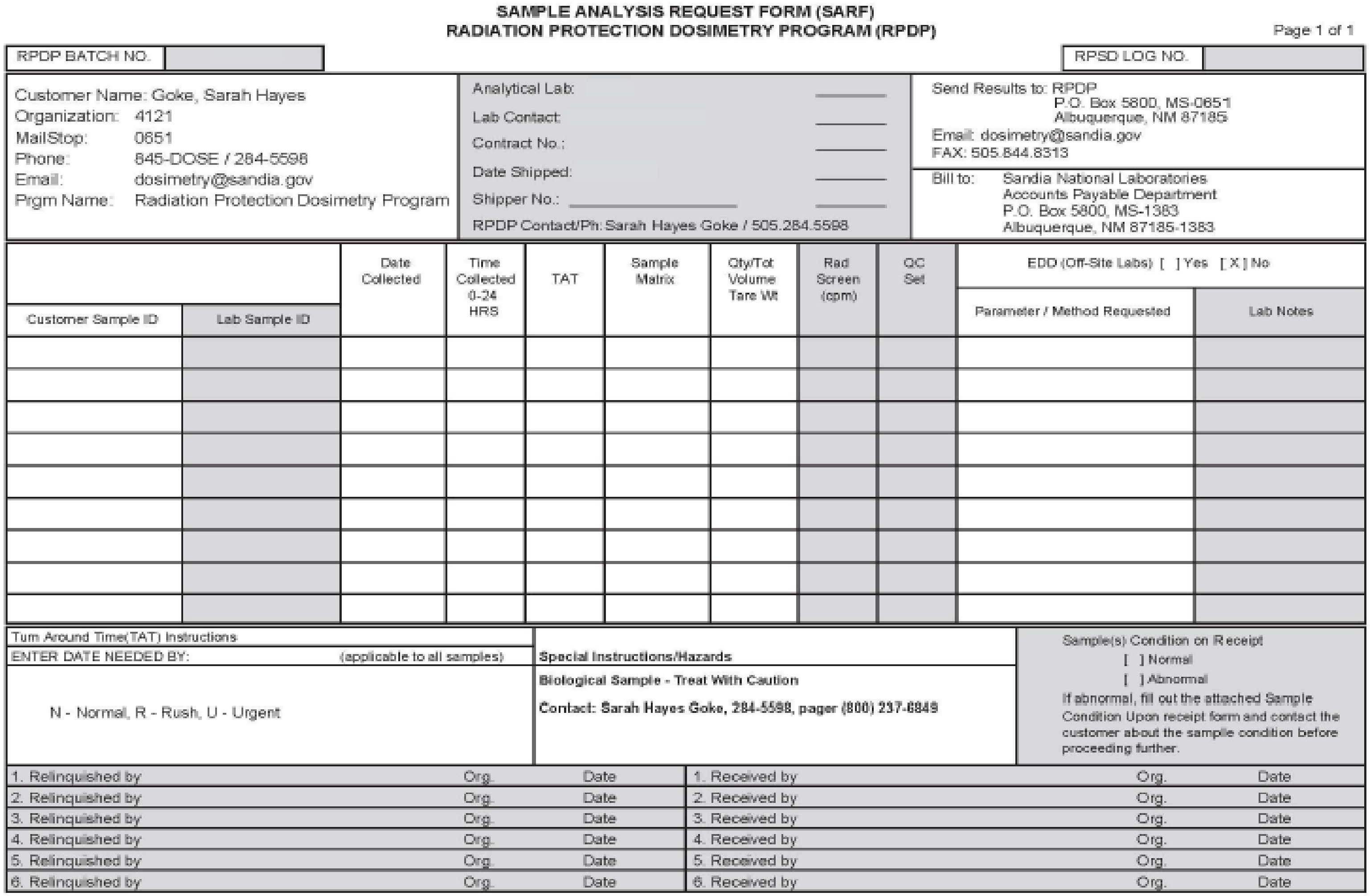
Samples Checked For
☐ Container Integrity
☐ Sample Size
☐ Sampling Label

Condition of Sample Receivec
☐ Acceptable
☐ Not Acceptable

Custody Seals
☐ Present
☐ Not Present

ATTACHMENT C
RPDP SAMPLE ANALYSIS REQUEST FORM

RPDP SARF



SAMPLE ANALYSIS REQUEST FORM (SARF)
RADIATION PROTECTION DOSIMETRY PROGRAM (RPDP)

Page 1 of 1

RPDP BATCH NO.

RPSD LOG NO.

Customer Name: Goke, Sarah Hayes
Organization: 4121
MailStop: 0651
Phone: 845-DOSE / 284-5598
Email: dosimetry@sandia.gov
Prgm Name: Radiation Protection Dosimetry Program

Analytical Lab:
Lab Contact:
Contract No.:
Date Shipped:
Shipper No.:
RPDP Contact/Ph: Sarah Hayes Goke / 505.284.5598

Send Results to: RPDP
P.O. Box 5800, MS-0651
Albuquerque, NM 87185
Email: dosimetry@sandia.gov
FAX: 505.844.8313

Bill to: Sandia National Laboratories
Accounts Payable Department
P.O. Box 5800, MS-1383
Albuquerque, NM 87185-1383

		Date Collected	Time Collected 0-24 HRS	TAT	Sample Matrix	Qty/Tot Volume Tare Wt	Rad Screen (cpm)	QC Set	EDD (Off-Site Labs) [ ] Yes [X] No	
Customer Sample ID	Lab Sample ID								Parameter / Method Requested	Lab Notes

Turn Around Time(TAT) Instructions
ENTER DATE NEEDED BY: (applicable to all samples)

N - Normal, R - Rush, U - Urgent

Special Instructions/Hazards

Biological Sample - Treat With Caution

Contact: Sarah Hayes Goke, 284-5598, pager (800) 237-6849

Sample(s) Condition on Receipt
[ ] Normal
[ ] Abnormal
If abnormal, fill out the attached Sample Condition Upon receipt form and contact the customer about the sample condition before proceeding further.

1. Relinquished by	Org.	Date	1. Received by	Org.	Date
2. Relinquished by	Org.	Date	2. Received by	Org.	Date
3. Relinquished by	Org.	Date	3. Received by	Org.	Date
4. Relinquished by	Org.	Date	4. Received by	Org.	Date
5. Relinquished by	Org.	Date	5. Received by	Org.	Date
6. Relinquished by	Org.	Date	6. Received by	Org.	Date

ATTACHMENT D: ONSITE LABORATORY (RPSD) ANALYSIS REQUEST & CHAIN-OF-CUSTODY

RPSD SARF

Sandia National Laboratories
Sample Analysis Programs

Sample Analysis Request Form
Page ____of ____

To be completed by Customer

Shaded areas are for Lab use only

Customer Name: ___________________
Customer Email ID: ___________________
Organization: ___________________
Phone: Sample ___________________
Location (Bldg/Rm): ___________________
Date Results Needed: ___________________
Project/Task Number: ___________________

Hazards/Special Instructions:

☐ Provide EDD

Batch Log No: _________________
Logged By: _________________

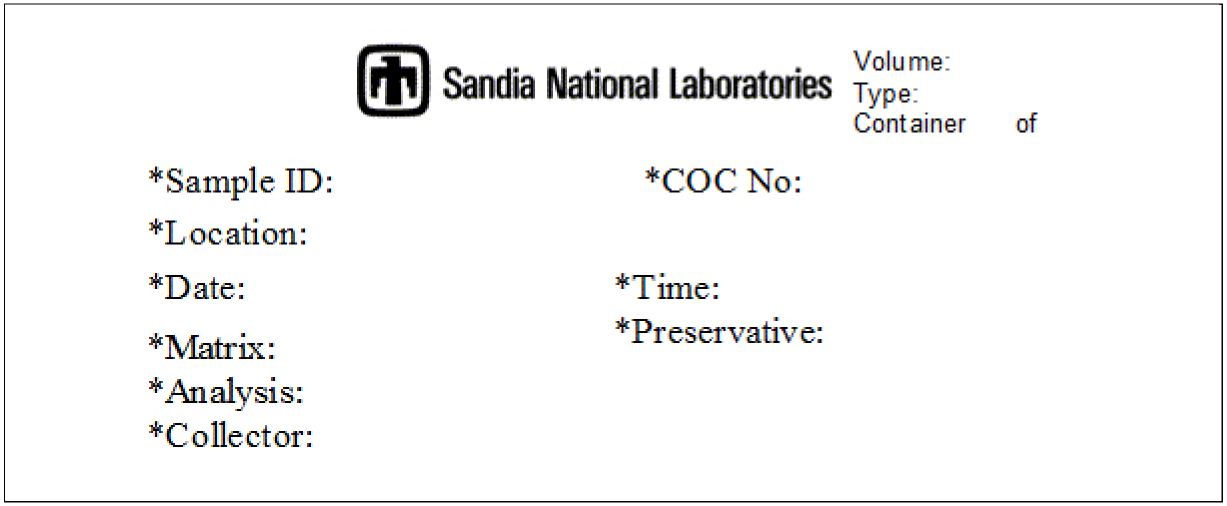
o RPOP - Rad Protection Operation
o RPID - Dosimetry
o RPSD - Sample Diagnostics
o IH - Industrial Hygiene
o DND -DeconDecom
o EXT- External
o SND - Source & Device
o WM – Waste Management
o OTH - Other

o EM - Environment Monitoring
o EMAA - Ambient Air
o EMWW - Waste Water
o EMGW - Ground Water
o EMTS - Terrestrial survell
o EMSW - Storm Water
o CMC – Coop Monitoring Ctr
o ER – Enviroment Restoration

Customer Sample ID	Sample Type	Date/Time Collected	Sample Amount or Flow Rate	Requested Analysis	Survey or COC#	Lab ID	Rad Screen(CPM)	Remarks/Aliquot Amount

Relinquished by ___________________ Date _____________________ Received by ________________________ Date_______________________
Relinquished by ___________________ Date _____________________ Received by ________________________ Date______________________

ATTACHMENT E
SAMPLE LABEL



Sandia National Laboratories

Volume:
Type:
Container of

*Sample ID: *COC No:
*Location:
*Date: *Time:
*Preservative:
*Matrix:
*Analysis:
*Collector:

* Required Fields

Attachment F: Radiological Survey Form

http://www-irn.sandia.gov/esh/radpro_procedures/forms/rsf.dot

Survey Number:

RADIOLOGICAL SURVEY FORM

Page: ________ of ________

Location:	Requester/Org.:	Date:	Time:

Purpose:	Request #:	RWP#:

Instrument and Probe Type and Serial Number		Surveyor(s) Printed Name(s)	Surveyor(s) Signature/Date

#	Item Description/Location	BETA-GAMMA ACTIVITY Counting Data Attached: ☐ YES ☐NO % Eff.: [e] _____ Radionuclide: _____				ALPHA ACTIVITY Counting Data Attached: ☐ YES ☐NO % Eff.: [e] _____ Radionuclide: _____				RADIATION SURVEY Background: ______ mrem/hr Radiation Type: Gamma	
		cpm	Bkg. cpm	dpm[a, b] / 100cm2	T/R/F[c]	cpm	Bkg. cpm	dpm[a, b] / 100cm2	T/R/F[c]	mrem/hr	Distance from Source[d]

[a] ND = No detectable activity above background [b] If other than 100 cm^2, indicate area or record as 'dpm/probe' or 'dpm/LAW' (large area wipe). [c] T/R/F = Total/Removable/Fixed [d] OC or CT = On Contact [e] %Eff-Removable/Direct

Remarks:		
	Reviewed by:	Date:

ATTACHMENT G: Sample Management Log

DATE	PROJECT NAME	RELINQUISHED BY	RCVD. BY	PARENT C.O.C.	SHIP DATE	DESTINATION LAB	SHIPPER #	WAYBILL #	<5 Y/N	QTY.	WGHT.	COMMENTS

ATTACHMENT H: DISCLAIMER

NOTE:

The samples in this cooler are labeled radioactive per Sandia National Laboratory policies and procedures.

However, the samples have been analyzed by gamma spectroscopy and are not radioactive materials per DOT regulations (49 CFR 173.403). Gamma spectroscopy results are enclosed with the chain-of-custody.

Signature:______________________________________ Date:______________________

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Page _____ of ______

AUTHORIZED USERS LIST

Document Title: Sample Management Office (SMO) Sample Handling, Packaging and Shipping Laboratory Operating Procedure

Document Number: LOP 94-03 **Revision:** 6

By my signature below, I affirm that I have read and understand this document, and all references called out in procedural steps, and I agree to operate within the stated constraints.

Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date
Name (printed)	Signature	Dept./Company	Date

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SANDIA NATIONAL LABORATORIES
LONG-TERM STEWARDSHIP

HEALTH AND SAFETY PLAN

PLA 05-09

GROUNDWATER MONITORING HEALTH AND SAFETY PLAN

Author: Robert Ziock **Date:** 1/10/12
Robert Ziock, Subject Matter Expert

Approved: Michael F. Skelly **Date:** 10 Jan 12
Michael Skelly, Project Leader

Approved: Don Schofield **Date:** 1/10/12
Don Schofield, Field Support Operations Project Leader

Approved: Pamela Puissant **Date:** 1/24/12
Pamela Puissant, Department Manager

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*]	*No*

EFFECTIVE DATE: 1/24/12

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Revision History

Revision	Review Date	Effective Date	Summary of Changes
1	9/29/2005	10/10/2005	New document
2	7/30/2007	8/16/2007	Formatting changes. Updated section 2, Roles and Responsibilities; section 3, Training Qualifications; section 11, References.
3	3/4/2010	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	1/10/2012	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.

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Table of Contents

TABLES

ATTACHMENTS

EP 2009-ALW – Environmental Planning – Activity Level Work Evaluation Form
EP 2009-AUL – Environmental Planning – Authorized Users List
EP 2009-OJT – Environmental Planning – On-the-Job Training
EP 2009-WRA – Environmental Planning – Work Resumption Authorization Form

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Acronyms & Abbreviations

ABC	Rating system for fire extinguishers. Class A extinguisher are for ordinary combustible materials; Class B extinguishers are for flammable or combustible liquids; Class C extinguishers are for electrical fires (extinguishing agent is non-conductive).
AOP	administrative operating plan
EOC	Emergency Operations Center
ES&H	Environment, Safety and Health
FOP	field operating procedure
GFCI	ground fault circuit interrupter
HASP	health and safety plan
HCl	hydrochloric acid
HNO_3	nitric acid
H_2SO_4	sulfuric acid
KAFB	Kirtland Air Force Base
LOP	laboratory operating procedure
LTES	Long Term Environmental Stewardship
MSDS	Material Safety Data Sheet
NaOH	sodium hydroxide
OJT	on-the-job training
OSHA	Occupational Safety and Health Administration
pH	potential of hydrogen
PHS	primary hazard screening
PLA	plan
PPE	personal protective equipment
ppm	parts per million
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
VOC	volatile organic compound

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1.0 Purpose, Goals and Objectives

Purpose	The purpose is to recognize and anticipate all potential hazards associated with performing groundwater monitoring activities at Sandia National Laboratories, New Mexico (SNL/NM).
Goals	The goal is to perform groundwater sampling activities with zero occupational injuries and reportable occurrences. The activities are described in detail in the associated technical work documents (TWDs) (i.e., administrative operating procedure [AOP], field operating procedures [FOPs], laboratory operating procedure [LOP], sampling analysis plan [SAP], and mini-SAP).
Objectives	The objectives are: • Perform work identified in the TWDs for groundwater sampling activities. • Plan this work so that potential hazards are recognized and controlled. • Perform health and safety protocols to prevent hazards to workers and protection of the environment. • Execute the work **only** as it is identified and described in the TWDs for groundwater sampling activities listed in section 3.0. The work shall be performed in a manner that protects personnel from hazards, thus preventing injury. • Limit work activities to authorized and trained personnel. • Improve this document and work processes (if necessary) based on feedback from personnel and lessons learned.

2.0 Roles and Responsibilities

The Long-Term Stewardship Department is responsible for development, approval, distribution, revision, and control of this document.

The **project leader** is responsible for the following:

- Reviewing and recommending approval of this procedure.
- Providing overall coordination and management of the project.
- Providing a SAP that meets prescribed regulatory or programmatic requirements.
- Reviewing completed field forms and data pertaining to waste management activities.
- Reporting all information as may be 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. It details sampling activities for the field support operations project leader and field technicians. It summarizes sampling procedures, analytical parameters, field measured parameters, purge requirements, and waste management tasks. It also 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 request to appropriate department (Hazardous Waste Management Facility Radioactive and Mixed Waste Management Facility, Waste Water, 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 project 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 for the on-the-job training (OJT) of new field technicians.
- Assigning field technicians (qualified by training and experience) to conduct the activities described in this procedure.

- Coordinating sampling activities with the sampling coordinator, Sample Management Office and field technicians.
- Ensuring the materials and equipment necessary to perform the work are available.
- Providing the field technicians with a copy of the mini-SAP.
- Reviewing field documentation.
- Maintaining the training matrix for all field personnel.
- 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.
- 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.
- Maintaining and decontaminating equipment.
- Managing and disposing of waste as directed by completed Work Request Forms, (FOP 05-04, Attachment A) and the field support operations project leader.
- Completing and reviewing field documentation forms.
- Providing recommendations for revisions to this plan (if necessary).

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 Environmental Stewardship (LTES) Groundwater Monitoring Activities*.
- Read and sign AOP 95-16, *Sample Management and Custody*.
- Read and sign laboratory operating procedure (LOP) 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, *LTES 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).
- 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)
*ENV102	OSHA Health & Safety Basic Training - General Worker (24 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
RAD230R	Radiological Worker II Retraining
RGH100	Crane, Rigging, Hoisting, & Hands-on Training

*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 health and safety plan (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

A task hazard analysis (THA) has been performed on all groundwater sampling activities in conjunction with a PHS SNL05A01241, *LTES 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 lists 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.

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)

An Activity Level Work Evaluation Form (EP 2009-ALW) was completed and approved by the department manager as required by AOP 09-10, *Work Planning and Control*. The completed form applies only to the activities as they are identified and described in the FOPs, AOP, and LOP listed in Section 3.0

A brief description of groundwater sampling activities is listed below and is followed by a THA table.

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
- Documentation of 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) Contaminated 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.	Medium	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. Notebook containing Material Safety Data Sheets (MSDSs) kept in sampling vehicle.
Mechanical (pinch points) 1) Motorized reel for raising and lowering pump. 2) Hydraulic lift on back of sampling vehicle.	Medium	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) (continued)

Potential Hazard	Hazard Rating	Control
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.	Medium	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 Valley Craft model number UG III-2K# 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

Any individuals have the authority to shutdown groundwater sampling 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)
    - Sever 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)

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 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

A variety of emergency situations could possibly occur during groundwater sampling activities. These include:

- Personal injury/illness
- Fire
- Meteorological events any other natural disaster
- Security incidents
- KAFB closures

Points of contact and Emergency Telephone Numbers are provided in Table 3.

Table 3. Points of Contact and Emergency Telephone Numbers

Resources and Contact	Telephone Number
SNL/NM Incident Command System (Fire, Ambulance)	911 (505-844-0911 from cell phone)
SNL/NM Medical Clinic	845-8159 or 844-0081
SNL/NM Non-Emergency Number	262-7222 (urgent care)
Poison Control Center	272-2222
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 Field Support Operations Project Leader Don Schofield	office: 844-4088 mobile: 259-7098
SNL/NM Project Leader Michael Skelly	office: 845-7697 pager: 540-5513

Table 3. Points of Contact and Emergency Telephone Numbers (continued)

Personnel to Notify If an Incident Occurs	
SNL/NM Department Manager Pamela Puissant	office: 844-3185 mobile: 239-9144
Industrial Hygiene/Safety Jeffrey Throman	office: 844-5779 pager: 283-1381

A First Aid kit is available on site to treat minor injuries. Minor injuries must be examined by the Sandia Medical Clinic.

Paramedic services can be dispatched through SNL/NM Incident Command System (911 or 844-0911).

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.

Sandia National Laboratories, Long-Term Stewardship, FOP 05-04, *Groundwater Monitoring Waste Management* (latest edition), SNL/NM.

Sandia National Laboratories, Long-Term Stewardship, FOP 09-05, *Conducting Slug Test Using Pressure Transducer and Data Logger* (latest edition), SNL/NM.

Sandia National Laboratories, Environmental Programs and Long-Term Stewardship, FOP 10-01, *Borehole and Downhole Well Video Inspection*, (latest edition), SNL/NM.

Sandia National Laboratories, Environmental Programs and Assurance Department, PHS SNL05A01241, *Long Term Environmental Stewardship Groundwater Monitoring Activities* (latest edition), SNL/NM.

SNLNM00825, *9925/1 (High-Bay)*, SNL/NM, September 2009.

SNLNM00827, *Groundwater Monitoring: Roving*, SNL/NM, September 2009.

SNLNM01481, *Groundwater Surveillance: Roving*, SNL/NM, February 2010.

SNLNM01520, *Chemwaste Landfill Groundwater Surveillance: Roving*, SNL/NM, February 2010.

Valley Craft UG III-2K# STD F89815A5, August 30, 2010

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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

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), department home page

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Attachment B

SNL Medical Facilities Location Map

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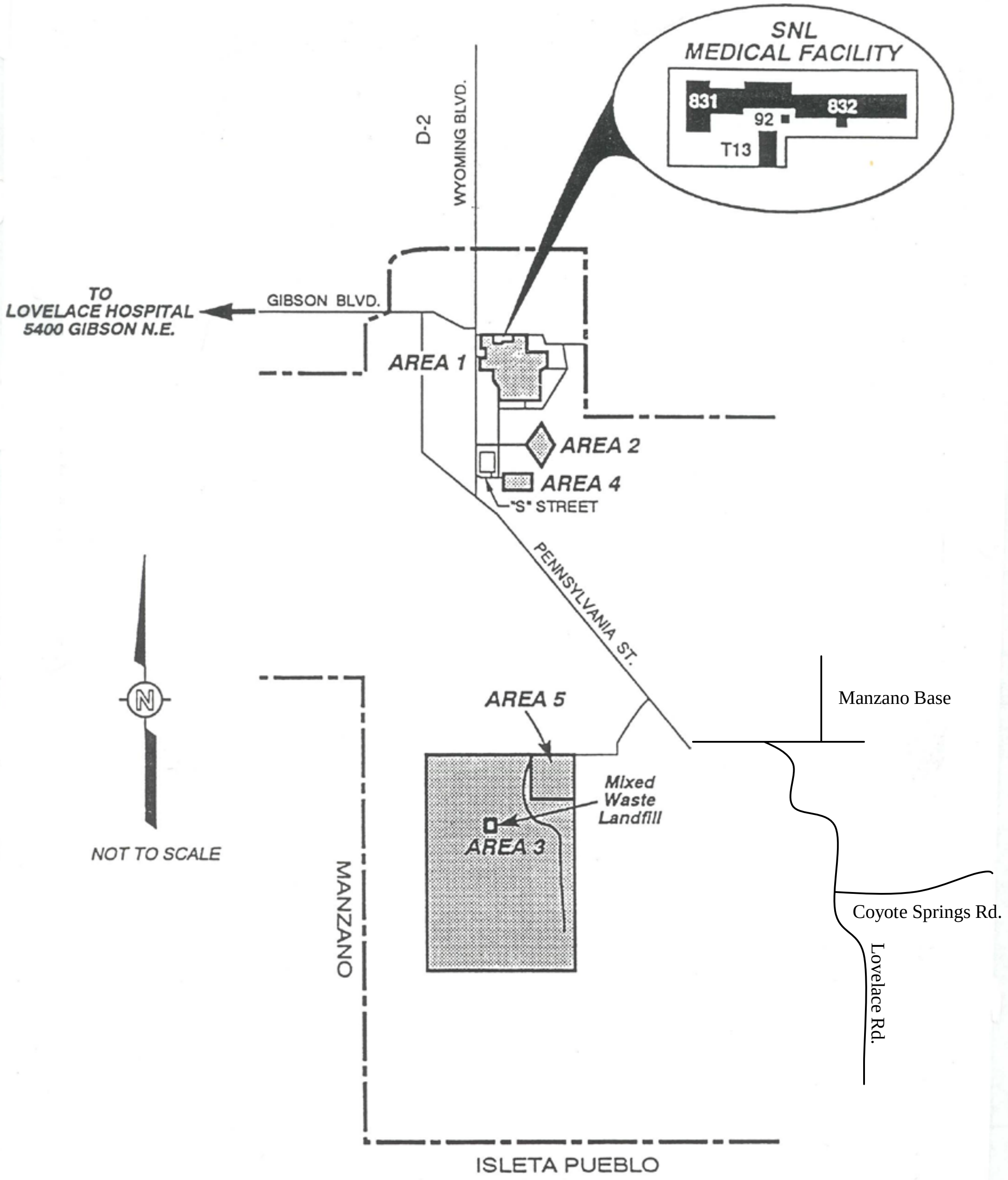
SNL
MEDICAL FACILITY
831
832
92
T13
D-2
WYOMING BLVD.
TO
LOVELACE HOSPITAL
5400 GIBSON N.E.
GIBSON BLVD.
AREA 1
AREA 2
AREA 4
"S" STREET
PENNSYLVANIA ST.
N
NOT TO SCALE
AREA 5
Mixed
Waste
Landfill
AREA 3
MANZANO
ISLETA PUEBLO
Manzano Base
Coyote Springs Rd.
Lovelace Rd.

SANDIA NATIONAL LABORATORIES
Sample Management Office (SMO) DEPARTMENT 4142

PROCEDURE FOR COMPLETING THE CONTRACT VERIFICATION REVIEW (CVR)

SMO-05-03
Revision 05

Approved: Wendy Palencia **Date:** 11.5.2013
Wendy Palencia, Author,
SMO Technical Support

Approved: **Date:** 11.05.2013
Lorraine Herrera, SMO Technical Support

Approved: Pamela Puissant **Date:** 11/12/13
Pamela Puissant, Manager

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: 11/12/13

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TABLE OF CONTENTS

LIST OF APPENDICES

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Revision History

Revision	Effective Date	Summary of Changes
Rev 1	June 9, 1999	New document, first issue
Rev 2	December 2, 2003	Technical changes
Rev 3	April 30, 2007	Technical changes
Rev 4	May 11, 2010	Edited sections due to method changes. Added section to cover the Data Anomaly Report (DAR). Renamed Issue to Revision.
Rev 5	November 12, 2013	Expanded section on processing the DAR. Added Responsible Individual descriptions, footnote disclaimer, and Acronyms and Abbreviations. Minor sentence and grammar edits. Added yes/no option for closure to Problem Resolution section of CVR form. Removed yes/no "Resolved" boxes on section 1 and 2 of CVR form.

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ACRONYMS AND ABBREVIATIONS

AOP	Administrative Operating Procedure
ARCOC	Analysis Request and Chain of Custody
brw	Oracle Query Builder File
CSV	Comma Separated Values file
CVR	Contract Verification Review
DAR	Data Anomaly Report
EDD	Electronic Data Deliverable
EPA	U.S. Environmental Protection Agency
GC/HPLC	Gas Chromatography/High Performance Liquid Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HRGC/HRMS	High Resolution Gas Chromatography/High Resolution Mass Spectrometry
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample
IDL	Instrument Detection Limit
L_c	Critical Level
LC/MS/MS	Liquid Chromatography/Mass Spectrometry/Mass Spectrometry
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MB	Method Blank
MDA	Minimum Detectable Activity
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NNSA/SFO	National Nuclear Security Administration/Sandia Field Office
OP	Operating Procedure
ppm	Parts Per Million
PQL	Practical Quantitation Limits
QAPP	Quality Assurance Project Plan
QC	Quality Control
RRT	Relative Retention Time
SA	Sample
SDG	Sample Delivery Group
SMO	Sample Management Office
SNL/NM	Sandia National Laboratories New Mexico
SOW	Statement of Work
STAR	Sample Tracking and Analytical Results
TAT	Turnaround Time
TIC	Tentatively Identified Compounds

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1.0 PURPOSE, SCOPE, AND OWNERSHIP

1.1 Purpose

This document provides instructions for performing a contract verification review (CVR) of analytical data packages received by the Sandia National Laboratories/New Mexico (SNL/NM) Sample Management Office (SMO). The CVR checks completeness and compliance of the sample custody and laboratory report documentation. Sample custody documentation is generated during a sampling event. The laboratory that performs the sample analyses generates the laboratory analytical report. The CVR determines whether or not sample custody is completely documented, and whether or not the laboratory complied with technical and reporting requirements of their contract.

The CVR examines specific items of sample management, custody, and laboratory reporting. The field sampling team and the analytical laboratory enter the sample management and custody items on the Analysis Request and Chain of Custody (ARCOC) form SMO 2012-ARCOC. After sample receipt at the laboratory, the laboratory completes the ARCOC and returns a copy to the SMO for sample login verification. Additionally, the complete ARCOC original is included in the final laboratory analytical report.

Contract verification review items checked in the laboratory analytical report are technical, quality control (QC), and reporting requirements imposed upon the laboratory through the *SNL/NM Contract Statement of Work (SOW) for Analytical Laboratories*. The laboratory is required to provide specific information in all analytical reports at the request of SNL/NM SMO. These data are often in addition to the laboratory's standard operating procedures or requirements of the analytical method. The CVR provides the SMO with a record of laboratory performance on a report deliverable basis, and allows for SMO tracking of reporting deficiencies, correction requests, and problem resolutions. The SMO monitors the performance of contracted laboratories, in part, using results from the CVR.

1.2 Scope

This procedure applies to all sampling and analysis events coordinated through the SNL/NM SMO and/or utilizing analytical laboratories under contracts administered by the SMO. Any sampling and analysis event, whether for environmental monitoring, site investigation, site restoration, waste characterization, industrial hygiene, bioassay or other purpose, which is tracked by the SMO and for which an SMO contract laboratory generates a data report, will be monitored by the SMO utilizing the CVR. The SMO is responsible for completing the CVR, initiating and tracking any corrective actions based upon the CVR, and documenting corrective action closure.

1.3 Ownership

The SNL/NM SMO is the owner of 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.

2.0 RESPONSIBLE INDIVIDUALS AND ORGANIZATIONS

The **Department Manager** is responsible for the following:

- Providing programmatic guidance leading to the development of this OP.
- Reviewing and approving this procedure.
- Acting as liaison to U.S. Department of Energy (DOE) and National Nuclear Security Administration/Sandia Field Office (NNSA/SFO) regarding sample management issues.

The **SMO Technical Lead** is responsible for the operations and activities conducted within the SMO. The principal responsibilities of the SMO Technical Lead include the following:

- Updating this procedure.
- Developing and maintaining the *SNL/NM Contract SOW for Analytical Laboratories*.
- Managing contractor laboratory services, including procurement, reviewing routine performance assessments, and conducting general laboratory oversight.

The **SMO QA Coordinator** is responsible for the following:

- 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.

The **Project Coordinator** is responsible for coordinating efforts associated with SMO analytical services. The principal responsibilities of the Project Coordinator include the following:

- Acting as a point of contact between Task/Project Leaders and the analytical laboratories.
- Obtaining appropriate sample containers from a vendor or analytical laboratory.

- Scheduling projects with contract laboratories.
- Notifying analytical laboratories of any quality assurance, environmental, safety, health, and sample matrix requirements regarding sample handling, preparation, and analysis.
- Resolving problems, issues, non-conformances, and errors for projects with regard to analytical data.
- Performing CVR to ensure appropriate QC analyses have been performed in accordance with the *SNL/NM Contract SOW for Analytical Laboratories*.
- Performing QC of data entered into the SMO database.
- Performing electronic data QC and transfer.
- Processing and follow-up on any data package corrections, both hardcopy and electronic.
- Providing technical guidance and information, as required.
- Reviewing, verifying, and processing proformas and invoices from contractors.

The principal responsibilities of the **Laboratory Oversight/Data Validation Contractor**, as reflected in the applicable contract, include the following:

- Performing laboratory oversight as directed by the SNL/NM SMO.
- Conducting visits to and technical system audits of, contractor laboratories to ensure compliance with *SNL/NM Contract SOW for Analytical Laboratories*.
- Performing data validation in accordance with the applicable procedures.
- Communicating non-compliance issues to the SMO Technical Lead and/or SMO Project Coordinator(s).
- Verifying implementation of laboratory corrective action plans.

3.0 PROCEDURES

3.1 Prerequisites and Associated Procedures

Prerequisite to completing the CVR, reviewers must be familiar with the appropriate requirement documents and associated procedures. SMO personnel shall read this procedure and sign the Authorized Users List, EP 2009-AUL. Applicable documents include current revisions of the following:

- *Quality Assurance Project Plan (QAPP) for the SNL/NM Sample* Management *Office*, SMO QAPP,

- The current analytical laboratory contract including the *SNL/NM Contract SOW for Analytical Laboratories*, and

- The project specific sampling and analysis plan and/or quality assurance project plan (or equivalent).

Associated procedures include current revisions of the following:

- *SMO Data Management Plan,* Administrative Operating Procedure (AOP) 95-44,
- *Procedure for Electronic Data Deliverable (EDD) Processing*, SMO-05-04,
- *Administrative Operating Procedure for Sample Management and Custody,* AOP 95-16, and
- *Data Validation Procedure for Chemical and Radiochemical Data SNL/SMO,* AOP 00-03.

3.2 Completing the CVR

The CVR will be completed on analytical data packages delivered to the SMO from the contracted analytical laboratories. The CVR must be completed prior to validation and forwarding to the sampling task manager and the Records Center. The CVR shall be maintained in the Records Center in accordance with the requirements in section 2.4.1 of the SMO QAPP. A copy of the CVR form can be found on the SNL Controlled Document site under SMO Forms, SMO 2012-CVR. Alternatively, the CVR may be completed and electronically signed and date stamped using the CVR tool at https://rails-rn-prod.sandia.gov/esh_smo/cvr/.

Upon receipt of the analytical data package, the SMO will log the Sample Delivery Group (SDG) number and receipt date into the Sample Tracking and Analytical Results (STAR) database, retrieve the current correspondence and documentation files associated with the specific sampling activity, and place the package in queue awaiting CVR.

The CVR is divided into six topical sections, with line entries to be checked as complete or incomplete under each section. The six sections are:

- 1.0 Analysis Request and Chain of Custody Record and Log-In Information
- 2.0 Analytical Laboratory Report
- 3.0 Data Quality Evaluation
- 4.0 Calibration and Validation Documentation
- 5.0 Data Anomaly Report
- 6.0 Problem Resolution

The electronic CVR Form can be found on: Q:\SMO\SMO-2012-CVR.doc.

A copy of the CVR form is provided in Appendix A.

To complete the CVR, gather the analytical data report including the original, signed, returned ARCOC, and the current correspondence and documentation file. Complete the following information.

Header Information

- Project Leader
- Project Name
- Project/Task Number
- ARCOC Number
- Analytical Lab
- SDG Number

The header information items will be found on the ARCOC and on the analytical laboratory report.

Review the items indicated on each line of the CVR. If the items indicated are complete and correct check "Yes" in the appropriate column. If the items are incomplete or incorrect check "No" and record an explanatory note. The reviewer should resolve any deficiencies with the documentation, if possible, during the review.

3.2.1 Review the ARCOC and laboratory login information and complete the CVR Section 1.0.

Line 1.1 All items on ARCOC complete – data entry clerk initialed and dated

All information prompted for on the ARCOC is necessary for accurate tracking of the samples and documenting sample custody. Verify that all items have been completed and that the sample custody record is complete and unbroken. Check that the SMO data entry clerk has initialed and dated the ARCOC (on the field copy) indicating when the sample data were entered into the STAR database.

Line 1.2 Container type(s) correct for analyses requested

Check that the recorded sample container types are compatible with the sample matrices and analyses requested. Recommended containers are listed in Attachment 5, *SNL/NM Contract SOW for Analytical Laboratories*.

Line 1.3 Sample volume adequate for # and types of analyses requested

Check that the volume or mass of sample provided meets or exceeds the analysis minimum requirement, including sufficient volume/mass required for quality control analyses.

Line 1.4 Preservative correct for analyses requested

Sample preservation, both chemical and thermal, must be consistent with guidance and requirements of the U.S. Environmental Protection Agency (EPA). Correct preservatives are found in Attachment 5, *SNL/NM Contract SOW for Analytical Laboratories*.

Line 1.5 Custody records continuous and complete

Check that a member of the sampling team listed on the ARCOC was first to relinquish the samples. There should be no time gaps in the custody record and all custodians should be identified, from sample collection until receipt at the laboratory. If an express carrier transported samples to the laboratory they should be identified and the shipment waybill number recorded.

Line 1.6 Lab sample number(s) provided and SNL sample number(s) cross referenced and correct

Laboratory sample numbers that uniquely correspond to the SNL/NM assigned sample numbers must be indicated on the returned ARCOC or sample acknowledgement. SNL/NM sample numbers must be correctly cross-referenced to the laboratory sample numbers in the analytical data package.

Line 1.7 Date samples received

The date that samples were received at the laboratory must be indicated on the ARCOC. The date received is noted in the laboratory's acknowledgement of custody.

Line 1.8 Condition upon receipt information provided

The laboratory should make notation as to whether or not the samples were received intact, in good condition, and correctly preserved. Any anomalies should be noted.

3.2.2 Review the analytical laboratory report and complete the CVR Section 2.0.

Line 2.1 Data reviewed, signature

Check that there is a transmittal letter or section in the laboratory report case narrative testifying to laboratory management's review and approval for release of the analytical data. The testimonial should bear the signature of an appropriate laboratory manager.

Line 2.2 Method reference number(s) complete and correct

The analytical methods used by the laboratory should be standard, published methods and reflect the latest promulgated revisions. EPA numbers will typically identify the methods in the report. Check that the analytical methods are referenced and conform to the ARCOC.

Line 2.3 QC analysis and acceptance limits provided (MB, LCS, and sample replicate)

Analytical laboratory batch QC sample analysis results and result acceptance limits must be provided in the report. The types of QC samples analyzed will depend on the analytical method and requirements of the *SNL/NM Contract SOW for Analytical Laboratories*. Typically, minimum batch quality control might consist of a method blank (MB), laboratory control sample (LCS), and laboratory control sample duplicate (LCSD). Sample replicates should not be performed on field QC samples. Sample replicates are required for most inorganic and radiochemical analyses.

Line 2.4 Matrix spike/matrix spike duplicate data provided

Organic method sample matrix spike analyses are run in duplicate on representative sample matrices, per batch, if adequate sample volume is provided. Check that the laboratory analyzed matrix spike/ matrix spike duplicate (MS/MSD) on the sample(s) and that accuracy and precision data in the sample matrix are reported. Inorganic and radiochemistry methods require MS only. MS should not be performed on field QC samples.

Line 2.5 Detection limits provided; PQL, MDL (or IDL), MDA and L_C

Limits of detection should be provided in the report for each sample analysis. The reported limits of detection should be appropriate to the analysis and in compliance with the *SNL/NM Contract SOW for Analytical Laboratories*.

Line 2.6 QC batch numbers provided

Check that analytical laboratory QC samples are identified by laboratory assigned batch numbers.

Line 2.7 Dilution factors provided and all dilution levels reported

Check that dilution factors are reported for all samples. Most samples will have a dilution factor of “1” indicating that the sample, digestion solution, or extract was analyzed using the optimum preparation weights and volumes described in the analytical method. All liquid samples, solid sample digestions, or extracts that may have required further dilution to be analyzed within the linear working range of the test instrumentation or standards curve must be identified and the dilution factor used must be provided.

Line 2.8 Data reported in appropriate units and using correct significant figures

The number of significant figures to be reported is specified in the *SNL/NM Contract SOW for Analytical Laboratories*. Typically, final analytical results may be reported at three significant figures and limits of detection reported at two significant figures. Check that results are reported in the appropriate units and with the correct number of significant figures. All data for inorganic and metal parameters shall be reported in parts per million (ppm).

Line 2.9 Radiochemistry analysis uncertainty (2-sigma error) and tracer recovery provided

Confirm that total measurement errors, expressed as plus and minus two standard deviations, i.e., 2-sigma, are reported for all final radiochemistry activity concentration results. Confirm the tracer recoveries are reported for alpha spectroscopy, and any other applicable radiochemical method.

Line 2.10 Narrative provided

Check that the analytical report narrative conforms to requirements in the *SNL/NM Contract SOW for Analytical Laboratories*. Generally, the report narrative will describe the contents of the data package, provide an index or list of analyses performed and samples processed, and describe the circumstances leading to laboratory qualification of any analysis result.

Line 2.11 Turnaround times (TAT) met

The analytical report is due at SNL/NM SMO after the elapsed time specified in the *SNL/NM Contract SOW for Analytical Laboratories* and indicated on the ARCOC (typically 30 calendar days from receipt of the samples). Mutually agreed upon expedited turnaround times may be applicable if documented and so noted on the ARCOC. Verify that the analytical report was received within the required turnaround time. Analytical data packages not meeting turnaround performance requirements may result in reduced or non-payment for services.

Line 2.12 Holding times met

Sample analysis holding times are counted as days from when the sample was taken until when it was prepared, if applicable, and analyzed. Analysis holding times may be required for valid analysis results or just recommended per industry guidance. Holding times for SNL/NM samples are listed in Attachment 5, *SNL/NM Contract SOW for Analytical Laboratories*. Analytical data not meeting holding times may result in reduced or nonpayment for services.

Line 2.13 Contractual qualifiers provided

The *SNL/NM Contract SOW for Analytical Laboratories* requires the laboratory to assign standard data qualifiers to analytical results not meeting specified criteria. For example, “B” will be assigned when sample analyte contamination was observed above the detection limit and in the associated batch preparation blank sample, and “J” will be assigned to indicate an estimated result less than a limit of quantitation but greater than a method detection limit. Check that the laboratory has appropriately assigned the correct data qualifiers. Qualifiers for SNL/NM samples are listed in Appendix G, *SNL/NM Contract SOW for Analytical Laboratories*.

Line 2.14 All requested result and Tentatively Identified Compounds (TIC) data provided

Confirm that all information requested from the laboratory on the ARCOC, in the *SNL/NM Contract SOW for Analytical Laboratories*, and in task specific correspondence was supplied.

3.2.3 Section 3.0 of the CVR, “Data Quality Evaluation,” provides opportunity to evaluate the analytical QC performance measures reported by the laboratory. Sample numbers are recorded to identify sample analysis results associated with nonconforming conditions or poor QC measurement results. Review line items in Section 3.0 highlight those laboratory reporting and technical performance measures checked for completeness in Section 2.0.

Complete the data quality evaluation, Section 3.0 of the CVR.

Line 3.1

Are reporting units appropriate for the matrix and meet SMO contractual specified or project-specific requirements? Inorganics and metals reported as ppm (mg/liter or mg/Kg)? Tritium reported in picocuries per liter with percent moisture for soil samples? Units consistent between QC samples and sample data?

Note the sample numbers for analytical results not meeting reporting unit conventions or requirements.

Line 3.2 Quantitation limit met for all samples

Note the sample number of analyses associated with elevated quantitation limits. Some analytical laboratories also call the quantitation limit the detection or reporting limit; for radiochemical analyses, the L_C or MDA are the limits reported. Quantitation limits will be elevated when high concentration of an analyte is present. This may only be a cause for concern when the other analytes are not detected in the same analysis.

Line 3.3 Accuracy

Note the sample numbers of analyses associated with nonconforming QC accuracy measurements. Analytical batch LCS analyte percent recoveries must fall within the specified control limits. Sample-specific surrogate compound percent recoveries (organic compound analyses) must be within established control limits. And, if required, matrix spike percent recoveries should also fall within acceptable limits.

Line 3.4 Precision

Note the sample numbers of analyses associated with nonconforming QC precision measurements. Precision measurement values are calculated as relative percent difference between the MS and MSD for organics, sample and sample replicate for inorganics, and paired LCS/LCSD for some analysis. Precision for radiochemistry is reported as the replicate error ratio.

Line 3.5 Blank data

Note the sample numbers associated with any blank sample in which significant positive results are observed. Contamination observed in a laboratory batch method blank sample, field or trip blank, or equipment rinse blank may indicate inadvertent or cross-sample contamination.

Line 3.6 Contractual qualifiers provided: "J"- estimated quantity; "B"- analyte found in method blank above the MDL for organic and inorganic; "U"- analyte undetected (results below the MDL, IDL or MDA (radiochemical)); "H"- analysis done beyond the method prescribed holding time; "h"- analysis done beyond the extraction/preparation holding time; "N" - result associated with spike analysis outside control limits.

Qualifier flags are to be assigned to analytical result data in accordance with requirements in the *SNL/NM Contract SOW for Analytical Laboratories*. Verify that laboratory qualifiers are correctly assigned and if not, record the affected sample numbers.

Line 3.7 Narrative addresses planchet flaming for gross alpha/beta

Check that the narrative includes information concerning planchet flaming for all gross alpha/beta analysis.

Line 3.8 Narrative included, correct, and complete

Check that information provided by the laboratory in the case narrative is accurate and in agreement with the analysis and QC data presented in the report. If the report narrative is inaccurate, note the sample numbers directly impacted.

Line 3.9 Second column confirmation data provided for methods 8330 (high explosives), pesticides/PCBs 8081 and 8082 and herbicides 8151.

Check that confirmation results data is provided for detected compounds for high explosive, pesticide/PCB and herbicide analyses.

3.2.4 Review the analytical data report to ensure it includes the required calibration and validation documentation and complete Section 4.0 for the CVR.

Line 4.1 GC/MS (8260 and 8270)

Verify the 12-hour tune check, initial calibration, continuing calibration, internal standard performance data, and instrument run logs are provided.

Line 4.2 GC/HPLC (8330, 8082, 9070A, and 8010)

Verify the initial calibration, continuing calibration and instrument run logs are provided.

Line 4.3 HRGC/HRMS (1668)

Verify the 12-hour tune check, initial calibration, continuing calibration, internal standard performance data, labeled compound recovery data, Relative Retention Times (RRTs) and ion abundance ratios for samples and standards, and instrument run logs are provided.

Line 4.4 LC/MS/MS (6850)

Verify the initial calibration, continuing calibration, CRI, internal standard performance data and instrument run logs are provided. For perchlorate analysis, verify that chlorine isotope ratios and ICS data are also provided.

Line 4.5 Inorganics (metals)

Verify the initial calibration, continuing calibration, ICP interference check sample data, ICP serial dilution, and instrument run logs are provided.

Line 4.6 Radiochemistry and General Chemistry

Verify the instrument run logs are provided.

Section 5.0 of the CVR, "Data Anomaly Report (DAR)," evaluates data results to historical monitoring data points to determine outliers and a need for data verification or sample reanalysis. The DAR is generated for monitoring and surveillance data.

3.2.5 Run the DAR and complete section 5.0 of the CVR.

Line 5.1 DAR completed for monitoring and surveillance data

When applicable, process the DAR according to steps below. The DAR shall be completed for all applicable sample data indicated as "SA", not field QC samples.

Update the Historical file

- Open Oracle Query builder
- Log in with user ID, password, "gspr1", press OK
- Click on "Open Query from File System"
- Press OK
- Open \\catbert\7500\SMO\STAR\SAR (suspected/data anomaly report) folder
- Select applicable browser file (i.e., SAR_Ambient_Filters.brw)
- Press "Open", then OK
- Execute query
- Maximize window
- Click on the "File" tab
- Select "Export Data"
- Select "Comma-Delimited (CSV)" as format
- Open "Select" window and open C:\SandiaSAR\SandiaSAR_Ver1p2\ applicable CSV file, press "Save", select "Yes" for "Save As"
- Press OK on export data window, then OK again
- Close file, press No

Run anomaly report

- Open SAR Application
- Press "Select EDD File"
- Go to \\catbert\7500\SMO\STAR\EDD by COC
- Highlight applicable EDD and press "Open"

- Press "Select Historical Data File" – select the CSV file you just updated under C:\SandiaSAR\SandiaSar_Ver1p2, then press "Open"
- Press "Load Parameters.xls"
- Highlight "parameters.xls", then press "Open"
- Press "Generate Report" – the report opens in a PDF format
- Right click on the PDF report and go to "Send to Mail Recipient"
- Select "PDF Format", then press OK
- Send Report to your mailbox and save in a file
- Press "Close Print Preview"
- Press "Generate SAR Workbook" – the report opens in an Excel format
- The Excel Report is automatically saved to your desktop under "SAR_ARCOC#"
- Review the report and note any anomalies on the CVR
- Notify task leader of any anomalies

3.2.6 Complete the CVR by noting the review results and correction tracking at the end of the form in Section 6.0 "Problem Resolution." Note any deficiencies and their resolution on the CVR, or initiate a nonconformance report or correction request. The Correction Request Form can be found on: Q:\SMO\Correction Request Form.doc.

A copy of the Correction Request Form is provided in Appendix B.

Record the date that any nonconformance report or correction request was forwarded to the laboratory. Sign and date the CVR. If corrections to the analytical report were required, then the CVR remains active and open until the corrected laboratory report is received. After receipt, review, and acceptance of the corrected analytical report, note closure of the corrective action by date and signature.

4.0 References

Sandia National Laboratories, *Administrative Operating Procedure for Sample Management and Custody*, AOP 95-16, current revision, Sandia National Laboratories, Albuquerque, New Mexico.

Sandia National Laboratories, *Data Validation Procedure for Chemical and Radiochemical Data*, AOP 00-03, current revision, Sandia National Laboratories, Albuquerque, New Mexico.

Sandia National Laboratories, *Procedure for Electronic Data Deliverable (EDD) Processing*, SMO-05-04, current revision, Sandia National Laboratories, Albuquerque, New Mexico.

Sandia National Laboratories, *Quality Assurance Project Plan for the SNL/NM Sample Management Office*, SMO QAPP, current revision, Sandia National Laboratories, Albuquerque, New Mexico.

Sandia National Laboratories, *SMO Data Management Plan*, AOP 95-44, current revision, Sandia National Laboratories, Albuquerque, New Mexico.

Sandia National Laboratories, *SNL/NM Contract SOW for Analytical Laboratories*, current revision, Sandia National Laboratories, Albuquerque, New Mexico.

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APPENDIX A
CONTRACT VERIFICATION REVIEW FORM

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Contract Verification Review (CVR)

Project Leader ____________________ Project Name ________________________ Project/Task No. ________________________

ARCOC No. ____________________ Analytical Lab ________________________ SDG No. ________________________

In the tables below, mark any information that is missing or incorrect and give an explanation.

1.0 Analysis Request and Chain of Custody Record and Log-In Information

Line No.	Item	Complete?		If no, explain
		Yes	No	
1.1	All items on ARCOC complete - data entry clerk initialed and dated			
1.2	Container type(s) correct for analyses requested			
1.3	Sample volume adequate for # and types of analyses requested			
1.4	Preservative correct for analyses requested			
1.5	Custody records continuous and complete			
1.6	Lab sample number(s) provided and SNL sample number(s) cross referenced and correct			
1.7	Date samples received			
1.8	Condition upon receipt information provided			

2.0 Analytical Laboratory Report

Line No.	Item	Complete?		If no, explain
		Yes	No	
2.1	Data reviewed, signature			
2.2	Method reference number(s) complete and correct			
2.3	QC analysis and acceptance limits provided (MB, LCS, Replicate)			
2.4	Matrix spike/matrix spike duplicate data provided			
2.5	Detection limits provided; PQL and MDL(or IDL), MDA and L_c			
2.6	QC batch numbers provided			
2.7	Dilution factors provided and all dilution levels reported			
2.8	Data reported in appropriate units and using correct significant figures			
2.9	Radiochemistry analysis uncertainty (2 sigma error) and tracer recovery (if applicable) reported			
2.10	Narrative provided			
2.11	TAT met			
2.12	Holding times met			
2.13	Contractual qualifiers provided			
2.14	All requested result and TIC (if requested) data provided			

Contract Verification Review (Continued)

3.0 Data Quality Evaluation

Item	Yes	No	If no, Sample ID No./Fraction(s) and Analysis
3.1 Are reporting units appropriate for the matrix and meet contract specified or project-specific requirements? Inorganics and metals reported as ppm (mg/liter or mg/Kg)? Tritium reported in picocuries per liter with percent moisture for soil samples? Units consistent between QC samples and sample data			
3.2 Quantitation limit met for all samples			
3.3 Accuracy a) Laboratory control sample accuracy reported and met for all samples			
b) Surrogate data reported and met for all organic samples analyzed by a gas chromatography technique			
c) Matrix spike recovery data reported and met			
3.4 Precision a) Replicate sample precision reported and met for all inorganic and radiochemistry samples			
b) Matrix spike duplicate RPD data reported and met for all organic samples			
3.5 Blank data a) Method or reagent blank data reported and met for all samples			
b) Sampling blank (e.g., field, trip, and equipment) data reported and met			
3.6 Contractual qualifiers provided: “J”- estimated quantity; “B”- analyte found in method blank above the MDL for organic and inorganic; “U”- analyte undetected (results are below the MDL, IDL, or MDA (radiochemical)); “H”- analysis done beyond the holding time; “h” - analysis done beyond the extraction/preparation holding time; “N” - result associated with spike analysis outside control limits			
3.7 Narrative addresses planchet flaming for gross alpha/beta			
3.8 Narrative included, correct, and complete			
3.9 Second column confirmation data provided for methods 8330 (high explosives), pesticides/PCBs 8081 and 8082 and Herbicides 8151			

Contract Verification Review (Continued)

4.0 Calibration and Validation Documentation

Item	Yes	No	Comments
4.1 GC/MS (8260, 8270) a) 12-hour tune check provided			
b) Initial calibration provided			
c) Continuing calibration provided			
d) Internal standard performance data provided			
e) Instrument run logs provided			
4.2 GC/HPLC (8330, 8082, 9070A, and 8010) a) Initial calibration provided			
b) Continuing calibration provided			
c) Instrument run logs provided			
4.3 HRGC/HRMS (1668) a) 12-hour tune check provided			
b) Initial calibration provided			
c) Continuing calibration provided			
d) Internal standard performance data provided			
e) Labeled compound recovery data provided			

f) RRTs for samples and standards provided			
g) Ion abundance ratios for samples and standards provided			
h) Instrument run logs provided			
4.4 LC/MS/MS (6850) a) Initial calibration provided			
b) Continuing calibration provided			
c) CRI provided			
d) Internal standard performance data provided			
e) Chlorine isotope ratios provided (perchlorate only)			
f) ICS provided (perchlorate only)			
4.5 Inorganics (metals) a) Initial calibration provided			
b) Continuing calibration provided			
c) ICP interference check sample data provided			
d) ICP serial dilution provided			
e) Instrument run logs provided			
4.6 Radiochemistry and General Chemistry a) Instrument run logs provided			

Contract Verification Review (Concluded)

5.0 Data Anomaly Report

Item	Yes	No	Comments
5.1 DAR completed for monitoring and surveillance sample data			
5.2 Problems or outliers noted			
5.3 Verification or reanalysis requested from lab			

6.0 Problem Resolution

Summarize the findings in the table below. List only samples/fractions for which deficiencies have been noted.

Sample/Fraction No.	Analysis	Problems/Comments/Resolutions

Were deficiencies unresolved? θ Yes θ No

Based on the review, this data package is complete. θ Yes θ No

If no, provide nonconformance report or correction request number ________________ and date correction request was submitted: ___________________________

Reviewed by: ______________________________ Date: ______________________

Were resolutions adequate and data package complete? θ Yes θ No

Closed by: ____________________________ Date: ____________________

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APPENDIX B
CORRECTION REQUEST FORM

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Date: ____________

To:	____________________	From:	____________________
Company:	____________________	Org:	____________________
Phone:	____________________	Phone:	____________________
Fax:	____________________	Fax:	____________________

Correction Request

COC: __________ SDG: ____________ Tracking No: __________

NOTE:

Sandia National Laboratories
Sample Management Office
P.O. Box 5800
Albuquerque, New Mexico 87185-1331

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SANDIA NATIONAL LABORATORIES
LONG TERM STEWARDSHIP DEPARTMENT

FIELD OPERATING PROCEDURE

FOP 08-22

SOIL VAPOR SAMPLING

Author: Robert Ziock, SME **Date:** 6/8/11

Approved: Don Schofield, Field Support Operations Project Leader **Date:** 6/9/11

Approved: Paul Freshour, Department Manager **Date:** 6/8/11

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*]	*No*

EFFECTIVE DATE: 06/09/11

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), department home page

Revision History

Revision	Review Date	Effective Date	Summary of Changes
00	1/26/2009	1/27/2009	Original Issue
01	5/27/09	5/27/09	Section 6.3 – "Quality Control Sample Equipment Setup and Sampling Procedure" added.
02	6/08/2011	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.

Table of Contents

Tables

Figures

Attachments

Acronyms

AR/COC	Analysis Request/Chain Of Custody
AOP	Administrative Operation Procedure
CAMU	Corrective Action Management Unit
CWL	Chemical Waste Landfill
ES&H	Environmental Safety & Health
ft	foot/feet
FOP	Field Operating Procedure
GFCI	ground fault circuit interrupter
Hg	mercury
In.	inch/inches
LTMMP	Long-Term Monitoring and Maintenance Plan
MWL	Mixed Waste Landfill
NMED	New Mexico Environment Department
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
SMO	Sample Management Office
SNL/NM	Sandia National Laboratories/New Mexico
TA-V	Technical Area V
TEDS	Training and Employee Development System
THA	task hazard analysis
VOC	Volatile Organic Compound

1.0 PURPOSE, SCOPE, AND OWNERSHIP

Purpose

The purpose of this field operating procedure (FOP) is to provide guidelines and procedures for soil vapor sampling at Sandia National Laboratories/New Mexico (SNL/NM). This procedure addresses the collection and analysis of soil vapor samples at different SNL/NM project sites. While the basic procedure for collecting soil vapor samples is the same for all locations, different sites are subject to different regulatory requirements. In addition, construction of the soil vapor monitoring systems also varies (i.e., well head, above ground enclosure, Primary Sub-Liner Monitoring Subsystem), which may cause minor changes to the general sampling procedure. 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 sampling activities at SNL/NM. SNL/NM sites where soil vapor samples are collected include the Corrective Action Management Unit (CAMU) containment cell, Chemical Waste Landfill (CWL), Mixed Waste Landfill (MWL), and Technical Area V (TA-V). Site-specific information is provided in Attachments A, B, C, and D for the CAMU, CWL, MWL, and TA-V, respectively.

If other SNL/NM sites are subject to soil vapor sampling in the future, additional attachments will be added to this FOP to address site-specific requirements and any variations to the general sampling procedure 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 Long-Term Monitoring and Maintenance Plan (LTMMP) (MWL).

The **Field Support Operations Project Leader** is responsible for the following:

- Coordinating with the Department Manager, Project Leader 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 Training (Attachment E) 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 Leader** 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 sampling events.
- Providing copies of the relevant sections of the site-specific permit and sampling and analysis plan (SAP) (CAMU and CWL) and LTMMP (MWL) 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 Leader. At a minimum, required training shall include the training defined in this FOP, site-specific permits (CAMU and CWL), and LTMMP (MWL).
- Maintaining requisite training status.
- Inspecting and maintaining equipment.
- Completing a tailgate safety briefing prior to each day’s sampling activities.
- Collecting and storing samples properly.
- Delivering samples to the Sample Management Office (SMO) in a timely manner, relative to analytical holding times.
- 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 Leader and the Project Leader.

- Providing recommendations for revisions to this procedure (i.e., process improvement feedback as appropriate).

3.0 TRAINING QUALIFICATIONS

Personnel conducting soil vapor sampling shall complete all training required to perform work under this FOP and in accordance with site-specific permits and LTMMPs:

- Read this procedure and attachments relevant to the sampling being conducted.
- Read any applicable site-specific training (i.e, SNL Primary Hazard Screening [PHS], Health and Safety Plan, etc.)
- Read applicable sections of site-specific permit and SAP (CAMU and CWL), LTMMP (MWL) and comply with the related training program requirements.
- Meet and document minimum requirements (CAMU, CWL, MWL) for a Field Technician (on file at the CAMU Administrative Trailer).
- Complete training courses listed in Table 1.
- On-the-Job Training (Attachment E) is required for trainees. This training shall be completed at the request of the Field Support Operations Project Leader by an experienced Field Technician working with the trainee and documented on the form provided as Attachment E.
- Sign Authorized User List (Attachment F).

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	ES&H Awareness
MCH200	Hand and Power Tool Safety
MED102	Standard First Aid
MED104	Heartsaver CPR
OTS101	Occupational Thermal Stress
PPE106	Personal Protective Equipment Training
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 shall be completed prior to soil vapor sampling 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 low for activities identified in this FOP.

A site-specific Activity Level Work Evaluation Form shall be completed and approved by the Department Manager as required by Administrative Operating Procedure (AOP) 09-10, *Work Planning and Control*. Blank forms are available in Appendix C of AOP 09-10.

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. A Tailgate Safety Briefing Form (Attachment G) shall be completed at the time of the briefing.
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 will immediately notify the Project Leader, the Field Support Operations Project Leader, and the Department Manager. The Field Technician will seek the assistance of the Project Leader and Field Support Operations Project Leader for the mitigation of the hazard and/or issue and the completion of a Work Resumption Authorization Checklist. The checklist is available in Appendix D of AOP 09-10, *Work Planning and Control*. The Department Manager must sign the completed checklist prior to the restart of work.

4.1 Task Hazard Analysis

Task Description

Soil vapor samples are collected from various SNL/NM sites (i.e., CAMU, CWL, MWL, TA-V) and are analyzed to determine levels of 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.

Table 2 – Task Hazard Analysis

Level of Protection—Level D Personal Protective Equipment (safety shoes/boots, safety glasses)

Potential Hazard	Hazard Rating	Control
• Chemical (various VOCs)	Low	• There will be no contact with contaminated soils during soil vapor sampling activities. Soil vapors will be monitored using a PID as part of the purging process. Historically VOC levels have been low (parts per million). Eating, drinking and smoking will not be permitted while performing soil vapor sampling 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)	Low	• 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. • A management approved pressure safety data package is in place for equipment used for soil vapor sampling.
Radiological	None	• CAMU – There are no radiological hazards specifically related to soil vapor sampling at the CAMU. • CWL – There are no radiological hazards specifically related to soil vapor sampling at the CWL. • MWL – There are no radiological hazards specifically related to soil vapor sampling at the MWL. • TA-V – Entry into TA-V requires training that discusses radiation hazards and alarms associated with facilities in TA-V, but there are no radiological hazards specifically related to TA-V soil vapor sampling.
Fire	Low	• Fire extinguishers will be located in mobile equipment.

5.0 EQUIPMENT AND MATERIALS

The equipment and materials required for performing soil vapor sampling activities are as follows:

- Analysis Request/Chain-of-Custody (AR/COC) forms and sample labels.*
- Logbook (if applicable).
- Field forms:
  - ➢ SUMMA® Canister Log (Attachment H).

  - ➢ Soil Vapor Sampling Log (Attachment I).
- 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.

6.0 PROCEDURES

Note: Prior to conducting sampling refer to site-specific Attachments in this FOP for additional information.

Soil vapor sampling 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 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.
    - 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.

6.2 Soil Vapor Monitoring Inspection

An inspection of the sampling equipment and each soil vapor monitoring location shall be performed as part of each sampling event. The inspection shall be documented on the Soil Vapor Monitoring Inspection Form provided in Attachment J. Deficiencies shall be noted and repaired within 60 days. Details regarding the repair work (who performed work, what was done, and when it was completed) shall also be documented on the form.

6.3 Equipment Setup and General Soil Vapor Sample Collection

Equipment Setup

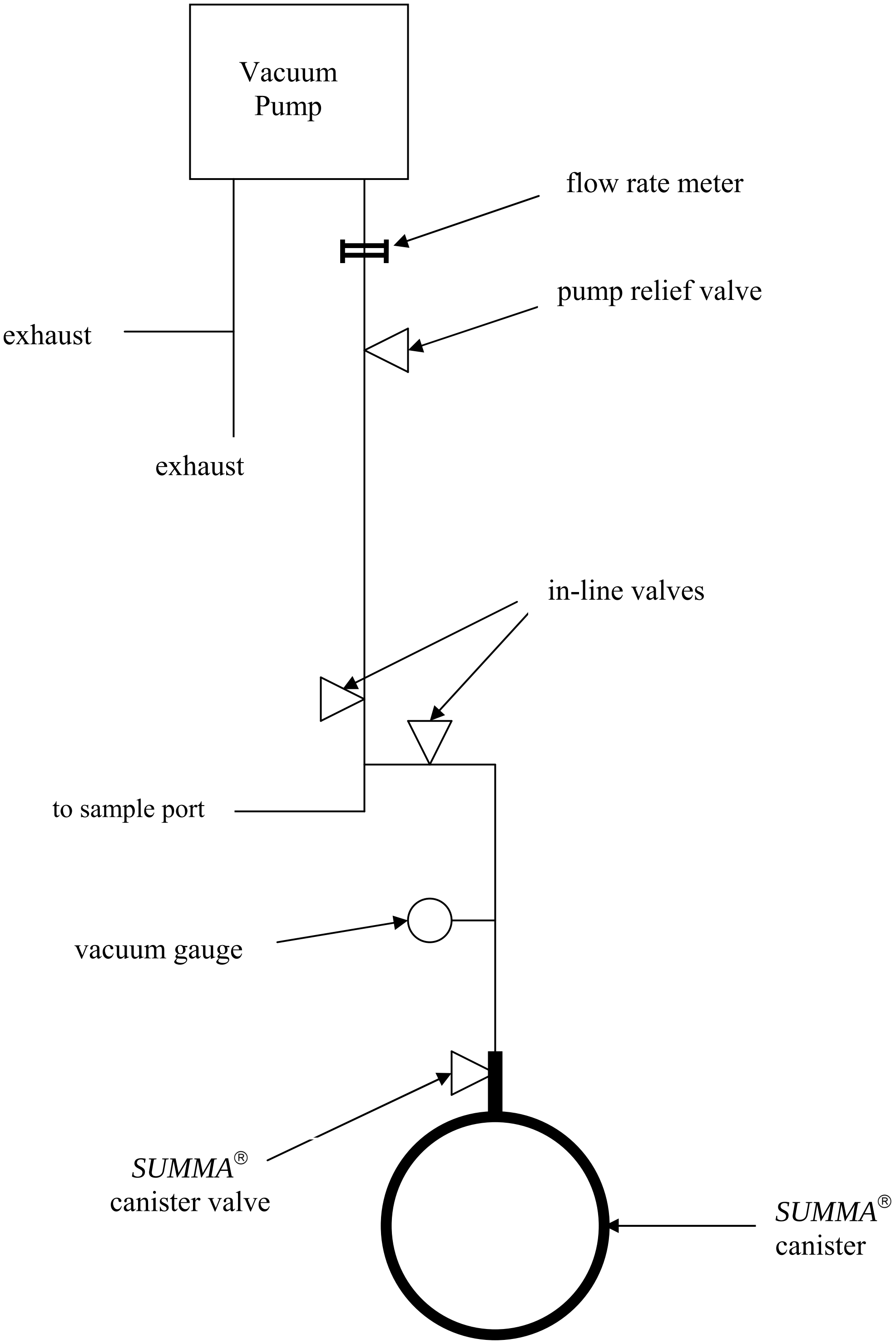
1) Load equipment detailed in Section 5.0 into sampling vehicle.
2) Position sampling vehicle adjacent to sampling location. The vehicle engine shall be turned off during purging and soil vapor sample collection.
3) Connect vacuum pump to AC power.

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 I).
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 H).
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.

6.4 Quality Control Sample Equipment Setup and Sampling Procedure

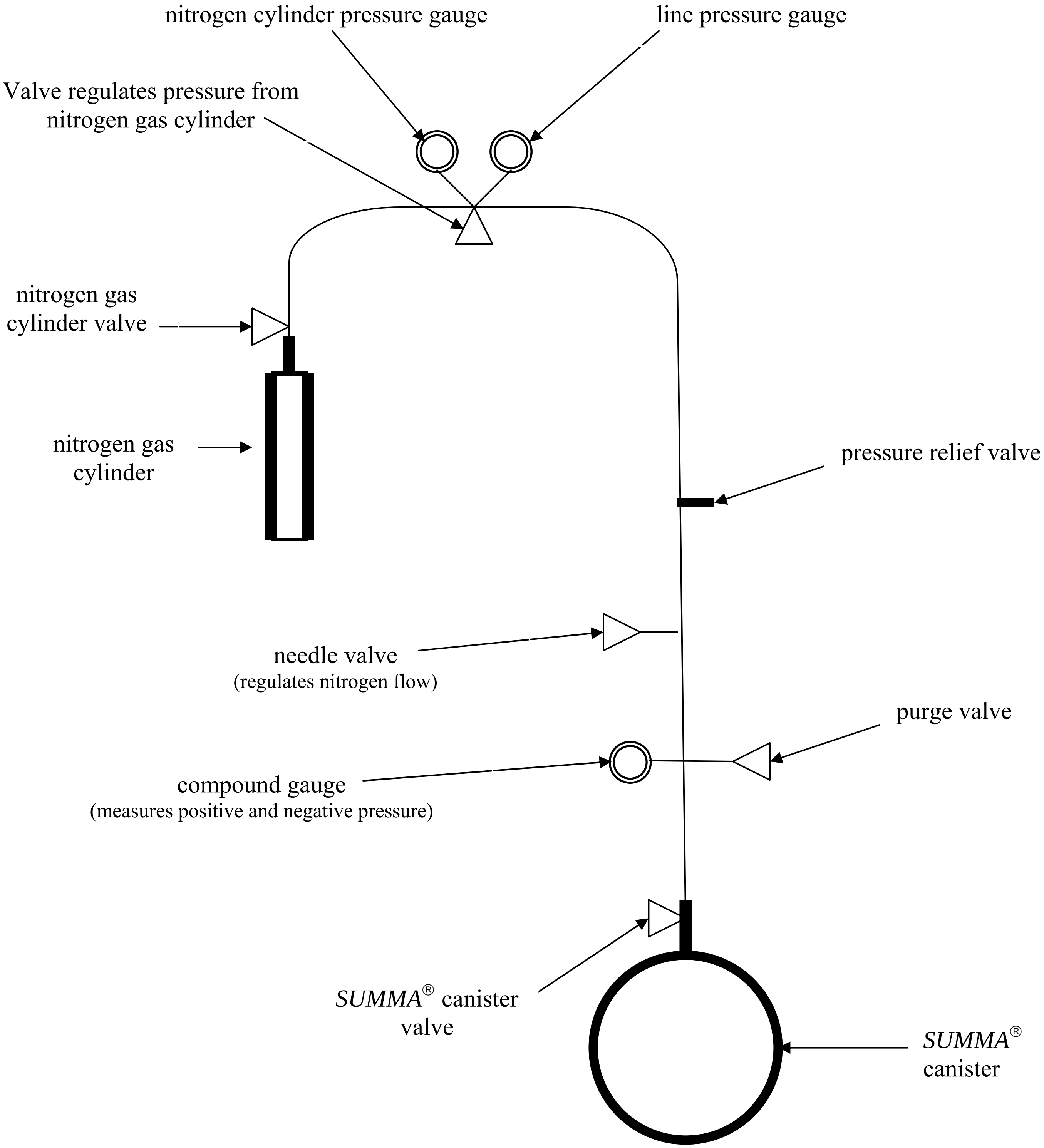
The site-specific SAP may require that a quality control sample of ultra pure nitrogen gas be collected in a SUMMA® canister. The quality control sample shall be kept in the presence of the other SUMMA® canisters during routine sample collection and will accompany the routine samples to the laboratory 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 valve.
2) Connect regulator manifold assembly to SUMMA® canister and cylinder containing nitrogen gas.
3) Open nitrogen gas cylinder valve.
4) Adjust regulator valve 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 valve.

Figure 6-4
Quality Control Sample Regulator Manifold and SUMMA® Setup



6.5 Shipping Samples to Laboratory

The SUMMA® canisters, AR/COC, and SUMMA® Canister Log will be taken to the SMO for shipment to the laboratory.

7.0 QUALITY ASSURANCE / QUALITY CONTROL

See site-specific quality assurance/quality control (QA/QC) guidelines and requirements as detailed in Attachments A through D and associated site-specific permits (CAMU and CWL) and LTMMP (MWL).

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 LTMMP (MWL). 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.

ES&H 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.

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 "Technical Area V Soil Vapor Well Sampling", SNL/NM (latest edition)

Attachment A

Corrective Action Management Unit

Site-Specific Information

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:

$$\text{V of soil vapor screen} = \pi * [(2\text{ in.})^2 / 4] * 12\text{ in.} * [1\text{ft}^3 / (12\text{ in.})^3] = 0.022\text{ ft}^3$$

$$\text{V of sampling tube} = \pi * [(0.25\text{ in.})^2 / 4] * 50\text{ ft} * (12\text{ in./ft}) * [1\text{ft}^3 / (12\text{ in.})^3] = 0.017\text{ ft}^3$$

Minimum pump running time to evacuate three volumes from each VSA sampling port is calculated as follows:

$$t = [(0.022\text{ ft}^3 + 0.017\text{ ft}^3) / (1.3\text{ ft}^3/\text{minute})] * 3 = 0.09\text{ minute or 5 seconds}$$

CSS Subsystem

Volume of the CSS tube (galvanized well pipe) are calculated as follows:

$$\text{V of galvanized pipe} = \pi * [(2.07\text{ in.})^2 / 4] * 21\text{ ft} * (12\text{ in./ft}) * [1\text{ft}^3 / (12\text{ in.})^3] = 0.49\text{ ft}^3$$

Minimum pump running time to evacuate three volumes from each CSS well is calculated as follows:

$$t = [(0.49\text{ ft}^3) / (1.3\text{ ft}^3/\text{minute})] * 3 = 1.13\text{ minutes or 68 seconds}$$

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.

$$\text{V of sampling tube} = \pi * [(0.17\text{ in.})^2 / 4] * 500\text{ ft} * (12\text{ in./ft}) * [1\text{ft}^3 / (12\text{ in.})^3] = 0.079\text{ ft}^3$$

Minimum pump running time to evacuate three volumes from the 500 ft. reel of tube is calculated as follows:

$$t = [(0.079\text{ ft}^3) / (1.3\text{ ft}^3/\text{minutes})] * 3 = 0.18\text{ minute or 11 seconds}$$

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

Attachment B

Chemical Waste Landfill

Site-Specific Information

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). 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 any work under the CWL PCCP, field technicians shall read the pertinent sections of the PCCP and complete/document all required training. Table B-1 summarizes important sections in the PCCP that pertain to soil-gas sampling.

Table B-1 CWL Permit Sections that Pertain to Soil Gas Sampling

Requirement	Permit Section	Comments
Monitoring and Records	Part 1, 1.8.7, p.8	Requirements for information that must be documented as part of the monitoring program. • The dates, exact place, and times of sampling or measurements; • The names of the individuals who performed the sampling or measurements; • The name and address of the laboratory that performed the analysis; • The dates analyses were performed; • The names of the individuals who performed the analyses; • The analytical techniques or methods used; and • The results of such analyses.
Record Keeping and Reporting	Part 2, 2.6, p. 15	Requirements for retention of all raw data, such as field forms, laboratory reports, and other supporting information gathered or generated during monitoring activities at the CAMU Administrative Trailer and/or the Record Center.
Soil Gas Monitoring - General	Part 3, 3.5, p. 26	General information on the CWL soil-gas sampling and analysis procedures and requirements that are described in Attachments 1 and 3 of the Permit
Personnel Training and Qualifications	Part 3, 3.6, p. 26	Requirement for compliance with the CWL-specific personnel training program detailed in Attachment 5 of this Permit.
Soil Gas Monitoring Process	Attach 1, 1.8.2.1 and 1.8.2.2, p. 39	General information on the CWL soil-gas sampling and analysis procedures and requirements.
Record Keeping – CAMU Admin Trailer & ESH Records Center	Attach 1, 1.12, p. 48-49; Attach 2, 2.21.4, p. 76-77; and Attach 3, 3.12, p. 90	Detailed information on record keeping requirements.
Soil Gas SAP	Attach 3 (p. 84-98)	Details CWL-specific sampling requirements.
Documentation of Inspections, Maintenance & Repair	Attach 4 implemented in Attach 1, 1.10 and 1.11, p. 45-48	Requirements for well and equipment inspections, maintenance, and repairs to be conducted as part of each annual sampling event (see Table 1-6).

Table B-1 CWL Permit Sections that Pertain to Soil Gas Sampling (concluded)

Requirement	Permit Section	Comments
Training Program	Attach 5, 5.0 p. 107-112	Job Titles, Qualifications & Roles and Responsibilities; Training Content, Frequency, and Techniques; Emergency Training, and Documentation Requirements all detailed in Attachment 5 of Permit.
Waste Types, Management & Spills	Attach 6, 6.0, p. 114	Hazardous waste generated at the CWL includes personal protective equipment (PPE) waste generated during the sampling of soil gas. Waste generated at the CWL will be stored and managed at the CAMU less-than-90-day waste accumulation area or another established less-than-90-day waste accumulation area.
Contingency Plan & Emergency Response	Attach 6, 6.4, p. 117-122	All field personnel must review the Contingency Plan and be familiar with its location in the CAMU Administrative Trailer and the basic emergency response process for the site.

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 in which these wells are screened. The D designation refers to "Deep" and is similarly indicative of the screen 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 procedure detailed in Section 6.0 of FOP 08-22 and Attachment 3 of the CWL PCCP.

Note that for the CWL, **a minimum purge time of 30 minutes per sampling port is required**. A minimum of 3 sampling tube volumes of air will be removed from each sampling port and at least 3 stable photoionization detector (or equivalent) readings shall be recorded prior to sample collection.

CWL Sampling Tube Volume and Purge Time Calculations

V of soil vapor screen = $\pi * [(0.31 \text{ in.})^2 / 4] * 2 \text{ ft} * (12 \text{ in./ft}) * [1\text{ft}^3 / (12 \text{ in.})^3] = 0.0010 \text{ ft}^3$
V of sampling tube = $\pi * [(0.215 \text{ in.})^2 / 4] * \text{tube length (ft)} * (12 \text{ in./ft}) * [1\text{ft}^3 / (12 \text{ in.})^3]$
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

Mixed Waste Landfill (MWL) Introduction and Status

In September 2007 the MWL Long-Term Monitoring and Maintenance Plan (LTMMP) was submitted to the New Mexico Environment Department (NMED) for review and approval. In December 2010 NMED indicated that the 2007 LTMMP would need to be revised and resubmitted after NMED approval of the Corrective Measures Implementation (CMI) Report. NMED approval of the CMI Report is pending, but anticipated in 2011. Once the CMI Report is approved, the LTMMP will be revised and submitted to NMED for review and approval. Soil vapor monitoring at the MWL will not be required until the LTMMP is approved. Based on the 2007 LTMMP and input from NMED, additional soil vapor monitoring wells will be installed at the MWL.

Once the LTMMP is revised and approved, this attachment will be updated to include the new well information and MWL-specific requirements. Information regarding the current soil vapor monitoring network at the MWL is provided below, but soil vapor sampling will not be required until the revised LTMMP is approved.

MWL Soil Vapor Sampling Network

The MWL soil vapor sampling network consists of two soil vapor monitoring wells, MWL-SV-01 and MWL-SV-02. The soil vapor implant at each location 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.

MWL Equipment Setup and Sampling Process

Follow the procedure detailed in Section 6.0 of FOP 08-22, *Soil Vapor Sampling*, and any applicable information in the approved version of the LTMMP.

MWL Sampling Tube Volume and Purge Time Calculations

V of soil vapor implant = $\pi * [(0.5 \text{ in.})^2 / 4] * 0.5 \text{ ft} * (12 \text{ in./ft}) * [1\text{ft}^3 / (12 \text{ in.})^3] = 0.0007 \text{ ft}^3$
V of sampling tube = $\pi * [(0.25 \text{ in.})^2 / 4] * 41 \text{ ft} * (12 \text{ in./ft}) * [1\text{ft}^3 / (12 \text{ in.})^3] = 0.0140 \text{ ft}^3$
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-1 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	1	0.0007	41	0.0140	0.015	0.044
MWL-SV-02	1	0.0007	41	0.0140	0.015	0.044

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 D

Technical Area V

Site-Specific Information

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

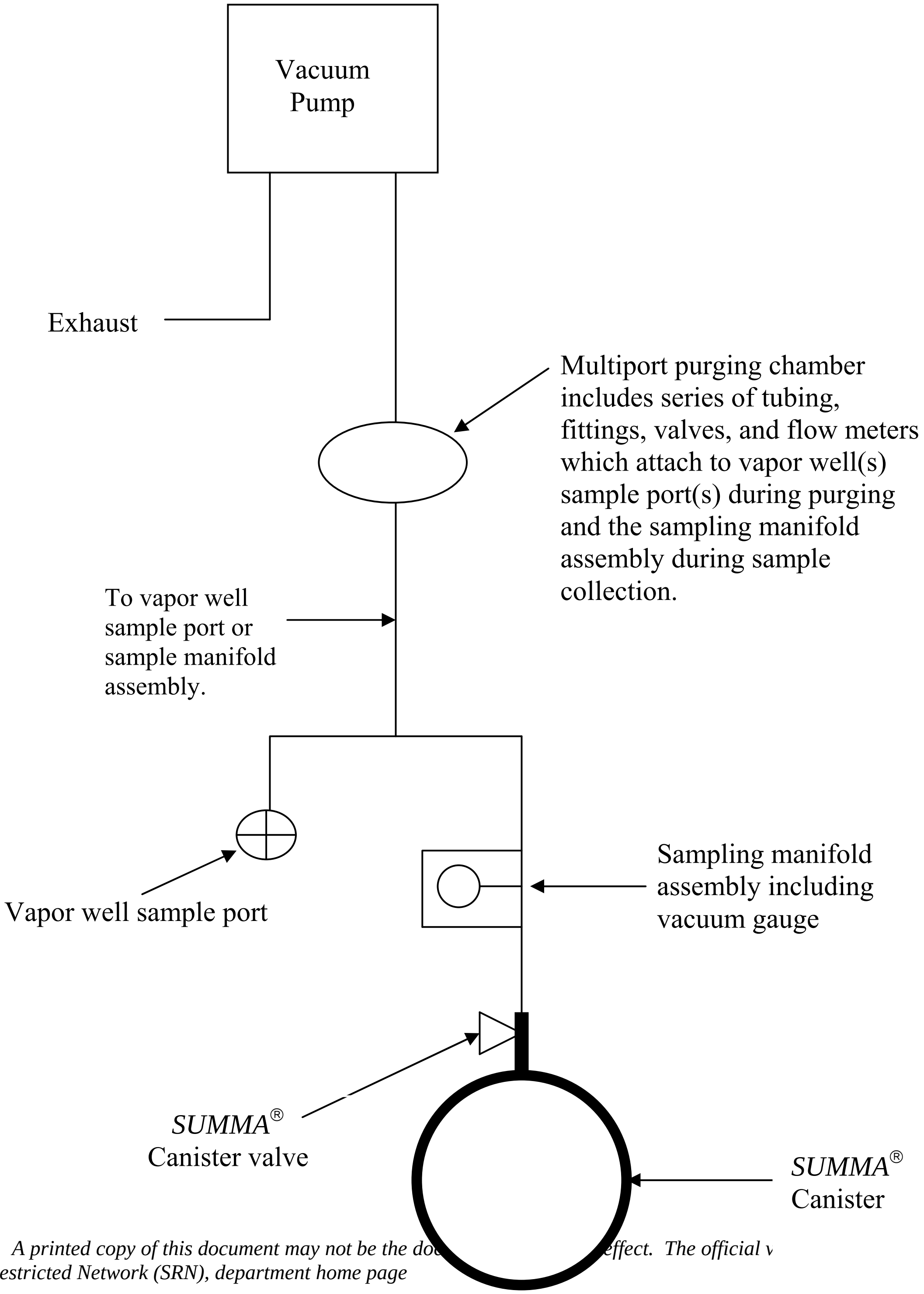
The TA-V soil vapor sampling equipment includes a sampling manifold assembly and a multiport purging chamber (Figure D-1). The multiport purging chamber is equipped with individual valves, fittings, and tubing which can be connected up to ten individual sample ports. The multiport purging chamber allows 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 I).
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 H).
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 = $\pi * [(0.5\text{ in.})^2 / 4] * 1\text{ ft.} * (12\text{ in./ft.}) * 1\text{ft.}^3 / (12\text{ in.})^3 = 0.0014\text{ ft.}^3$
V of sampling tubing = $\pi * [(0.25\text{ in.})^2 / 4] * \text{tubing length (ft.)} * (12\text{ in./ft.}) * [1\text{ft}^3 / (12\text{ in.})^3]$
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 E

On-The-Job Training

On-The-Job Training

By signature below, I affirm that the following is correct. Only those undersigned are authorized to conduct sampling as described in this plan.

Name of Trainee:	Signature	Org. / MS	Phone
Name of Trainer:	Signature	Org. / MS	Phone
Project:	**Date/Time In**	**Date/Time Out**	

Job Description:

Job Description (continued):

Equipment:

Additional Comments:

Date/Time In	**Date/Time Out**	Training Equipment/Experience

Job Description:

Job Description (continued):

Equipment:

Additional Comments:

On-The-Job Training (continued)

Date/Time In **Date/Time Out** Training Equipment/Experience

Job Description:

Job Description (continued):

Equipment:

Additional Comments:

Date/Time In **Date/Time Out** Training Equipment/Experience

Job Description:

Job Description (continued):

Equipment:

Additional Comments:

Date/Time In **Date/Time Out** Training Equipment/Experience

Job Description:

Job Description (continued):

Equipment:

Additional Comments:

Attachment F

Authorized User List

Authorized User List

By signing below, I affirm that I have read and I understand this procedure, and that I agree to operate within the stated constraints. Only those undersigned are authorized to conduct the activities described in this procedure.

Name (printed) Project Leader	Signature	Date	Org.
Name (printed) Field Technician	Signature	Date	Org.
Name (printed) Field Technician	Signature	Date	Org.
Name (printed) Field Technician	Signature	Date	Org.
Name (printed) Field Technician	Signature	Date	Org.
Name (printed) Field Technician	Signature	Date	Org.
Name (printed) Field Technician	Signature	Date	Org.
Name (printed) Field Technician	Signature	Date	Org.
Name (printed) Field Technician	Signature	Date	Org.
Name (printed) Field Technician	Signature	Date	Org.

Attachment G

Tailgate Safety Briefing

TAILGATE SAFETY BRIEFING

Facility: ____________________ Date: __________ Time: ______

Activities: ____

(Anyone has the right to cease field activities for safety concerns. The buddy system will be used when needed.)

Safety Topics Presented

☐ Wear safety glasses as needed	☐ Wear leather gloves when needed
☐ Wear safety boots	☐ Be aware of electrical hazards
☐ Wear hearing protection as needed	☐ Use safe lifting practices
☐ Be aware of biohazards (snakes, spiders, etc.)	☐ Be aware of slips, trips, and falls
☐ Perform appropriate inspections for equipment used	☐ Eating or drinking allowed in designated areas only
☐ Proper housekeeping will be maintained	☐
☐	☐

Does anyone have any weight restrictions on lifting? Circle **YES** or **NO**. If answered **YES** explain.

Presenter

Printed Name ____________ Signature ____________

Attendees

Printed Name ____________ Signature ____________

Printed Name ____________ Signature ____________

Printed Name ____________ Signature ____________

Printed Name ____________ Signature ____________

Printed Name ____________ Signature ____________

Notes

Attachment H

SUMMA® Canister Log

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

Attachment I

Soil Vapor Sampling Log

Soil Vapor Sampling Log

Location	Date	Time	Canister #	PID (ppm)	Starting Vacuum (in. Hg)	Ending Vacuum (in. Hg)	Location Comments

Attachment J

Soil Vapor Monitoring Inspection Form

Page 1 of 3

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.				

Page 2 of 3

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**

Page 3 of 3

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:

Inspector's Signature ___________________________
Original to: Site's Operating Record
Copy to: Environmental Operations Records Center, MS-1309

Sandia National Laboratories/New Mexico (SNL/NM)
Statement of Work
for Analytical Laboratories

Revision 6
January 2013

SANDIA NATIONAL LABORATORIES/NEW MEXICO (SNL/NM)
STATEMENT OF WORK FOR ANALYTICAL LABORATORIES

GENERAL INORGANIC, ORGANIC, RADIOCHEMICAL, BIOASSAY, IH, ASBESTOS, AND GEOTECHNICAL LABORATORY ANALYSES

Introduction

The Sandia National Laboratories (SNL) Sample Management Office is responsible for acquiring analytical services in support of SNL site activities. This statement of work (SOW) outlines the requirements for analytical services provided to Sandia Corporation by contract laboratories.

Samples obtained for chemical analysis in support of SNL activities will consist of soil, waste, groundwater, surface water, domestic supply water, air filters, demolition debris, biota, sludge, oil, organic liquids, swipes, organic and inorganic solids, air, soil gas, Industrial Hygiene (IH), and bioassay samples. In addition, samples may be acquired for airborne asbestos, bulk asbestos, or geotechnical testing. The sections below detail specific quality assurance protocols, analytical practices and procedures, analytical quality control requirements, deliverable formats, and schedule requirements. Collectively, these conventions have been established to ensure that Sandia data quality objectives are met and that data obtained from different contract laboratories are comparable.

Contract laboratories shall provide services that require processing samples with constituents including various types of chemicals and/or radioactive isotopes. Samples may include mixed waste (containing both Resource Conservation and Recovery Act hazardous and radioactive materials). Laboratories performing chemical analyses under this SOW must hold State of Utah Certification. Radioactive samples will be classified predominately as low-level waste; classification for transportation will be non-radioactive or limited quantity. Additionally, it is desirable for laboratories to have the capability to accept and analyze higher activity samples such as transuranic (TRU) waste or waste that would be classified as Type A radioactive material for transportation purposes. Laboratories must be able to comply with NVO-325 requirements per the Nevada Test Site Waste Acceptance Criteria (NTSWAC).

Laboratories' capabilities must include, at a minimum, general inorganic, organic, and radiochemical analyses. In addition, bioassay, asbestos, and geotechnical analyses may be included, or any combination thereof. Labs having IH or bioassay analysis capabilities shall perform the analyses in the primary facility or a sister laboratory within the same company. Bioassay laboratories must provide services to a U.S. Department of Energy (DOE) Laboratory Accreditation Program (DOELAP) approved site. Use of secondary laboratories shall not be permitted for bioassay analyses. Secondary laboratories will be considered for asbestos, TO-14, and geotechnical-type analyses.

Contract laboratories shall perform all analyses of samples received and obtain express written permission before sending any samples to a secondary laboratory for analysis. Prime contract laboratories, secondary, and sister laboratories may be required to successfully pass a DOE Consolidated Audit Program (DOECAP) systems audit and submit analysis results for performance evaluation samples prior to providing analytical support. Secondary and sister laboratories are required to show that they meet the requirements of this SOW. In the event that a secondary laboratory is approved and does receive samples, all delivery schedules shall remain unchanged.

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Section **Page**

• Attachment 1	General inorganic parameters and Target MDLs, PQLs
• Attachment 2	Radiochemical Parameters and Contract Required L_c, MDAs
• Attachment 3	Organic Parameters and Target MDLs
• Attachment 4	Geotechnical Test Methods
• Attachment 5	Holding Times and Preservation Techniques
• Attachment 6	Target Surrogate Recovery Control Limits
• Attachment 7	Analytical Request and Chain of Custody Form
• Attachment 8	AMP Policy for Payment Reduction NNSA Service Center Sites
• Attachment 9	AMP Policy for Information Sharing NNSA Service CenterSites
• Attachment 10	Routine Soil Sub-Sampling Procedure
• Attachment 11	SNL/SMO Electronic Deliverable Specification

1.0 ANALYSIS REQUESTS AND SAMPLE SHIPMENT

1.1 Work orders

Approximately one week before the start of sample collection, the Sandia Corporation Delegated Representative (SDR) will notify the contractor in writing of the scheduled shipment of samples. The notification shall take the form of an electronic mail bottle order issued against the contract. The bottle order will include sampling project information, anticipated project schedule, number of samples to be submitted, sample matrix, and anticipated analysis request to be performed by the contractor. Some small projects and/or rapid analysis requests may be submitted without the advance notification discussed above in order to meet project requirements. For emergency and expedited sample processing the SDR will notify the laboratory prior to sample shipment.

1.2 Shipping charges

Sample shipping charges to contract laboratories shall be paid by Sandia.

The contractor shall be responsible for shipping charges related to return samples and bottle orders.

1.3 Itemized analytical charges

Unit prices provided by laboratories shall include the cost of all Quality Assurance(QA), Quality Control (QC), preparation, extraction, cleanup, analytical, reporting, storage, and disposal requirements specified in this statement of work (SOW). Field QC samples, such as field blanks, field duplicates, and trip blanks, shall be invoiced and paid for at the itemized prices for field samples. Accelerated turn-around times are discussed in Section 4.2.1 of this SOW.

1.4 Analyte definitions

For analyses performed under this SOW, the term “general inorganic” refers to the analytes listed in Attachment 1, the term “radiochemical” refers to the analytes listed in Attachment 2, the term “organic” refers to the analytes listed in Attachment 3, and the term “geotechnical” refers to the tests listed in Attachment 4.

1.5 Time definitions

References to days, weeks, or months are defined as calendar days, weeks, and months unless otherwise specified. Report delivery schedules are discussed in Section 4 of this SOW.

1.6 Request for reanalysis

1.6.1 Incomplete reports and errors

If a suspected analytical error is identified by comparison with historical data, if QC data are either missing or outside the control limits, or if the data are unusable for any reason, the SDR reserves the right to request delivery of the missing documentation or the reanalysis of any or all samples within the sample lot. Where some results in the suite have met the acceptance criteria, reanalysis requests will be for the affected parameters only, rather than the entire analytical suite.

1.6.2 Reimbursement for reanalysis costs

Reanalysis will be requested by telephone or e-mail. Payment for reanalysis requested by the SDR shall be made according to the following guidelines:

a) Reanalysis requested because failed QC data were reported to the SDR shall not be paid for.

b) Reanalysis that are requested because of a suspected significant error, and to confirm the original results within reasonable analytical error, shall be paid for by Sandia. The SDR will seek input from the laboratory regarding reanalysis confirmation in light of sample inhomogeneity or other special considerations.

c) Reanalyses that are requested because of a suspected significant error, and that indicate that an analytical or reporting error was made in the first analysis, shall not be paid for by Sandia.

1.7 Nonstandard analyses

1.7.1 Bids for nonstandard analyses

Sandia may find it necessary, on occasion, to request an analysis that is not explicitly covered in this SOW. When this occurs, requests for quote will be submitted to each laboratory with a description of the needed work. If one or more of the laboratories holding current contracts can perform the analysis, the laboratory selection will be made based upon the prices submitted, an assessment by the SDR of the laboratory's ability to meet the technical specifications, and the capacity of the laboratories submitting bids. The contracts for laboratories having the needed capability will then be amended by letter to include the new analysis.

1.7.2 Proposing secondary laboratories

Laboratories shall obtain express written permission before sending any samples to a secondary laboratory for analysis. If none of the laboratories, holding current contracts, have the capability to perform the needed test, laboratories will then be allowed to propose secondary laboratories outside the current contract structure. If one or more of the proposals is accepted, the laboratories shall be solely responsible for executing a contract with the proposed laboratory for the work. The SDR will assess the need to perform an audit of the proposed laboratory prior to submitting samples. However, the laboratories under contract must ensure that all the applicable requirements of this SOW are met in lower-tier contracted work. When an audit is deemed necessary, failure to submit to or pass the audit will disqualify the proposed laboratory.

1.7.3 Using secondary laboratories

Laboratories shall not be permitted to send Sandia samples to laboratories outside the original contract structure unless the conditions described in Sections 1.7.1 and 1.7.2 have been met.

1.7.4 Deliverable levels

Sandia data deliverables routinely provided by the laboratory will be “Level C” reports. Laboratories will provide “Level D” and “Level B” reports upon request, and will provide line-item pricing for their preparation in the schedule of charges submitted to the Sandia National Laboratores/Sample Management Office SNL/SMO. Reporting definitions and analytical data deliverable requirements are fully outlined in Section 4 of this document.

2.0 QUALITY ASSURANCE REQUIREMENTS

2.1 General data quality objectives

2.1.1 Methods, quality control, and documentation

a) The SDR will sometimes find, through application of the U.S. Department of Energy (DOE) Streamlined Approach for Environmental Restoration (SAFER) process for data quality objectives (DQOs), that the QC or other requirements in this document should be relaxed or tightened to suit particular project needs. Individual project needs that necessitate requirements different from those discussed in this SOW will be negotiated on a case-by-case basis by the SDR subject to the limitations in the clause titled Delegation of Authority. However, any changes affecting cost must be approved by the Sandia Contract Representative.

b) DQOs are developed by the Sandia project specifically. However, a general requirement is that industry-standard methods, such as USEPA SW-846 (Third Edition, as revised and updated), USEPA 600 series methods, Occupational Safety and Health Administration (OSHA) methods, American Society for Testing and Materials (ASTM) methods, and American Public Health Association (APHA) methods (Standard Methods) be used where possible. The analytical requests submitted to a laboratory will generally specify which methods apply. In the absence of specific direction from Sandia, laboratories must employ a suitably sensitive analytical method that meets all project-specific quantitation levels. Laboratories should assume that (1) soil and waste samples should be analyzed using SW-846 methods, (2) aqueous samples should be analyzed using methods approved under the Clean Water Act, (3) air filters should be analyzed by approved National Institute of Occupational Safety and Health (NIOSH) methods or equivalent methods, and (4) tissue samples should be analyzed using SW-846 methods, with samples prepared as specified in Section 3.2.2 (g) of this SOW. Where industry-standard methods do not address particular analytes, performance-based methods may be utilized with prior approval from the SNL/SMO. All QC requirements specified in this SOW shall be met, even if they exceed those specified in a particular requested method.

c) Laboratories must prepare complete documentation for every activity in order to facilitate review and enhance defensibility of the data. Documentation requirements include records for sample receipt/login, preparation, digestion, extraction, sample or extract cleanup, standards preparation, and sample analysis.

d) In cases for which the specific QA and QC protocols found in this SOW, the DOE Quality Systems for Analytical Services document, and Chapter 5 of the National Environmental Laboratory Accreditation Conference (NELAC) standard cannot be extended to requested parameters, professional judgment shall be employed in adhering as closely as possible to the intent of those protocols. This means that the laboratory should extend all standard documentation and QC practices to parameters, methods, and analytical techniques that are not covered in these documents, where possible. The laboratory shall formulate an approach to performance and documentation of analytical procedures in light of the fact that it is the general goal of Sandia to obtain legally and technically defensible data. Specific QC and analytical requirements are discussed in detail in Section 3 of this SOW.

2.1.2 Certification and accreditations

Unless specifically allowed exemption by the SNL/SMO, laboratories serving Sandia must be accredited by NELAC and the State of Utah, and shall have successfully passed a DOE Consolidated Audit Program (DOECAP) audit. Laboratories performing analyses in support of the Sandia industrial hygiene programs must be American Industrial Hygiene Association (AIHA)-certified, and laboratories supporting personnel monitoring programs must be DOE Laboratory Accreditation Program (DOELAP)-approved.

2.2 Laboratory quality assurance plan

2.2.1 Specific requirements

The laboratory, and any secondary laboratories accepted for participation in the contract, shall have a laboratory quality assurance plan (LQAP) that contains sections or references addressing all of the items listed below.

a) Title page with provision for approval signatures and dates of revision.

b) Table of contents.

c) Laboratory organizational structure and key personnel responsibilities.

d) Personnel training, with required training, frequency, and methods of records maintenance specified.

e) Sample receipt, custody, and management practices. This section shall specify a formal vehicle for notifying the analytical group of holding times near expiration in order to minimize occurrences of expiration prior to analysis.

f) Facilities and equipment, including a description of security procedures, sample storage practices, and a list of equipment available at the laboratory. Equipment lists shall include acquisition dates.

g) List of all laboratory analytical procedures by method number and matrix. Laboratory policy shall require that controlled copies of analytical procedures be available to the analysts.

h) General instrument calibration and calibration verification policies, including documentation of calibration standards, coefficients resulting from linear or higher order polynomial regression calculations, calibration curve correlation coefficients, and the associated acceptance criteria. The issues below shall be addressed as applicable to the type of analyses being performed.

   i. Procedures shall require that linear regression calibration curve correlation coefficients (r) for general inorganic chemistry be ≥ 0.995.

   ii. Conformance with organic chemistry method calibration requirements shall be required. If linear regressions are used in calibration for organic methods, the LQAP or standard operating procedures (SOPs) must specify a minimum correlation coefficient or coefficient of determination of 0.99. If higher order polynomials are used, laboratories must obtain prior approval from the SDR and must follow the applicable guidance in SW-846 Method 8000.

   iii. Calibration frequency, methodologies, and documentation practices for radiochemistry counting instruments shall be discussed.

   iv. Evaluation practices for non-zero intercepts shall be addressed. Procedures shall require that linear and quadratic curves for both organic and inorganic data have initial calibration Y-intercept absolute values ≤3 times the method detection limit (MDL).

i) MDLs for general inorganic chemistry and organic chemistry. - The section addressing MDLs shall specify detection limit determination methodologies and shall include both empirical MDL verification and examination of the method blank populations for each analyte. Minimum MDL study requirements are discussed in greater detail in Section 3.3.1 of this SOW. Minimum detection amount (MDA) calculation requirements for radiochemistry are given in Section 3.3.4 of this SOW.

j) Default criteria for QC sample type, analysis frequency, data acceptance, and corrective actions for failures in daily QC practices. - Where QC acceptance criteria are not given in this SOW or in the applicable method, laboratory acceptance criteria shall be established statistically with a minimum of 20 data points by QC type, method/technique, and matrix. Control limits shall be established at the three-sigma (3σ) confidence level. Where data are insufficient to statistically establish acceptance criteria, an approved fixed limit may be temporarily applied. This section shall also discuss the QC data review processes employed by the laboratory. Laboratories performing radiochemical analyses shall specify default minimum tracer and carrier recovery criteria in the LQAP. QC data requirements and acceptance criteria for Sandia work are discussed in detail in Section 3 of this SOW.

k) A description of the corrective action report (CAR) process. - A copy of a CAR form shall be provided in the LQAP. The process shall include tracking and documentation of completion of all corrective actions.

l) A description of the laboratory document-control procedures, including archiving and archive retrieval.

m) An outline of the process for data review and approval. - Provision shall be made for peer, supervisory, or QA review of all chemist worksheets. In addition, the LQAP shall outline document flow, including review steps, from chain of custody (COC) to the final analytical report.

n) A discussion of the laboratory's holding time policies and processes for pre-preservation of sample bottles, sample preservation checks, and documentation of preservation checks. - Holding times and preservation techniques for Sandia samples are outlined in Attachment 5.

o) A discussion of the frequency and method of conducting and documenting internal audits. - In addition, the LQAP shall specify the frequency and contents of QA reports to laboratory management.

p) A list of approvals and certifications from states and external agencies. - The LQAP shall specifically require client notification when accreditations or certifications are revoked or suspended.

q) Specific laboratory policies regarding rounding and the number of significant figures to be used in reporting analytical results. Also, the LQAP or an SOP shall require that leading zeroes be used for numbers less than one and that units accompany all numbers that are not dimensionless. (The significant figures requirements for this SOW can be found in Section 4.1.13.) Additionally, the LQAP or an SOP shall define appropriate error correction practices and require the use of indelible ink for records.

r) A description of procedures for material procurement, quality inspection, inventory, and storage.

s) A discussion of methods for verifying the agreement of electronic data files with hard copy reports, including both electronic data deliverables (EDDs) and electronic reproductions of reports.

2.2.2 Standard Operating Procedure support for the Laboratory Quality Assurance Plan

The LQAP sections addressing some of the issues listed above may refer to detailed SOPs. Complete and comprehensive descriptions of all the listed processes are not required in the LQAP when the specific process details are outlined in SOPs. However, the supporting SOPs should be referenced in the LQAP.

2.3 Performance evaluation sample analysis requirement

2.3.1 Schedule

If requested to do so, chemical analysis laboratories shall perform the analysis of Sandia performance evaluation (PE) samples provided to the laboratory by the

SNL/SMO. The analytical and deliverable requirements for these PE samples are the same as all Sandia samples. Payment for the analysis of PE samples shall be made according to the fees specified in the contract. Sandia will not pay for the analysis of “known” samples provided as a courtesy for QC, investigations, or method development.

2.3.2 Performance Evaluation sample analysis

The analytical techniques and SOPs used in the analysis of PE samples shall be the same as those used in routine analysis of Sandia samples.

2.3.3 Proficiency

a) The SDR may provide a summary of analytical results and theoretical values for each PE round to each laboratory after all the data for that round is completed. Any requests for CARs necessitated by laboratory PE sample failures will accompany the summary report. Initial responses to CAR requests, including the projected schedule for completion, shall be due no later than two weeks from the date of the request. The SDR reserves the right to request accelerated delivery of CARs if circumstances make this necessary. Failure to respond promptly to a request for corrective action may result in temporary suspension of the laboratory from the SDR chemical analysis program.

b) Laboratory performance information may be shared among the National Nuclear Service Administration (NNSA) Service Center facilities and entities supporting NNSA Service Center site activities. The NNSA Service Center Analytical Management Program (AMP) policy governing the sharing of contractor performance information is provided as Attachment 5.

2.4 Systems and internal audit requirements

2.4.1 Annual systems audits

The laboratory shall undergo a DOECAP quality systems audit at least once a year. DOECAP audits will serve as a key performance indicator for verifying or denying the acceptability of an analytical laboratory to provide analytical services for DOE. Acceptability will be based primarily on the ability of the laboratory to produce data of known, adequate, and consistent quality. A formal audit report will be issued by the DOECAP following this activity. Responses to audit reports will be submitted to the DOECAP as specified in the DOECAP requirements documents.

An SDR systems review may be conducted at the laboratory at the discretion of Sandia. The purpose of this review is to verify laboratory compliance with the LQAP and the specifications of this SOW. In addition, recommendations may be made to laboratory personnel regarding possible quality improvements in light of good laboratory practices and/or industry standards. A formal report or request for corrective action may be issued following this activity. In that event, responses will be due 30 days from the date of issue unless a more rapid turnaround is necessary to safeguard data quality.

2.4.2 On-site data package review

Data package reviews may be conducted at the laboratory at the discretion of Sandia. The focus of these reviews shall be to verify contract compliance and deliverable accuracy, ensure that raw data and supporting documentation are maintained in retrievable form, and review ancillary documentation not included in deliverables. The data package to be reviewed will be chosen at the time of the review activity. A formal report or corrective action request may be issued following this activity. In that event, responses will be due 30 days from the date of issue.

2.4.3 Internal audits

The laboratory shall perform internal QA audits at least annually. A summary of the corrective actions resulting from the laboratory's internal QA audits shall be provided to the SDR in the quarterly progress report (QPR). QPRs are discussed in Section 2.14 of this SOW.

2.5 Participation in interlaboratory comparison studies

2.5.1 Required intercomparison programs

Laboratories performing inorganic or organic analyses shall participate in two single-blind, single-concentration performance testing (PT) studies per year, where available, for item (a) below. Laboratories performing radiochemical analyses shall participate in two single blind, single-concentration PT studies per year, where available, for item (b) below. Laboratories performing airborne silica, asbestos, metals, and/or organics analyses shall participate in item (c) below. Laboratories performing lead in paint analyses shall participate in item (d) below. Laboratories performing National Pollutant Discharge Elimination System analyses shall participate in item (e) below.

a) Commercial vendor programs designed to meet the requirements given in the Proficiency Testing section (Chapter II) of the NELAC standard.

b) Mixed Analyte Performance Evaluation Program (MAPEP), U.S. DOE, Idaho Operations Office, Idaho Falls, Idaho.

c) Proficiency Analytical Testing (PAT) Program, AIHA.

d) Environmental Lead Proficiency Analytical Testing Program (ELPAT), AIHA.

e) Discharge Monitoring Report--Quality Assurance Study (DMR-QA), U.S. Environmental Protection Agency (EPA) Office of Enforcement and Compliance Assurance.

2.5.2 Reporting intercomparison results

The laboratory shall report results of the intercomparison studies specified in Section 2.5.1 of this SOW to the SDR, quarterly. This report is due with the delivery of the laboratory QPR. All results received by the laboratory since the last quarterly report and more than one week before the due date shall be included in this deliverable. Results received less than one week before the due date may be held for inclusion in the next quarter's deliverable. Failure to participate in and report the results for the applicable intercomparison studies may result in suspension of the laboratory from the SDR laboratory analysis program.

Laboratories shall report all results from the participation of any approved PT program.

2.6 Employee training and documenting employee proficiency

The SDR is conscious of the value and worth of experience. Years of analytical experience may often gain equivalency to or outweigh academic achievement. It is required that laboratories have an internal analyst proficiency evaluation policy that provides a vehicle to gauge and document the competence of experienced individuals and specifies additional training and documentation practices applicable to all personnel. This policy shall include specific pass/fail criteria used for evaluations. Personnel that have not been trained and evaluated shall not participate in the handling or analysis of Sandia samples.

Evidence files must exist to demonstrate that each employee has met the laboratory’s minimum training requirements and has read, has understood, and is using the latest version of the laboratory's quality documentation. Training on specific equipment, analytical techniques, and laboratory procedures shall be documented.

Evidence must also exist to demonstrate that each employee has studied and acknowledged his or her personal ethical and legal responsibilities, including the potential penalties for improper, unethical, or illegal actions.

Laboratory personnel who are involved in receiving, processing, and/or managing Sandia samples shall be trained in radiation safety practices and techniques.

2.7 Laboratory instrumentation, equipment, and reagent maintenance

2.7.1 Instrument logs and response checks

a) The laboratory shall have an SOP that specifies the requirements for maintaining logbooks. These requirements shall specifically address QA protocols for error correction, as well as schedules for peer, supervisory, or QA review of logbooks. In addition, the use of indelible ink to make logbook entries shall be explicitly required.

b) The laboratory shall maintain an instrument logbook for all major instruments (excluding pH meters, conductivity meters, and the like) used to acquire data for the SNL/SMO. Each instrument logbook shall be clearly labeled to indicate its association with a particular piece of laboratory equipment.

c) Laboratories performing general inorganic analysis of Sandia samples shall have an SOP requiring that instrument response checks, or other appropriate instrument performance checks, be performed daily. The requirements shall include recording the results of such checks in the associated instrument maintenance log.

d) Laboratories performing organic analysis of Sandia samples shall have an SOP requiring that instrument logs contain a brief description of run failures and the file names for analysis runs. Reanalysis run entries, including those for necessary dilutions, shall reference the original run to facilitate review. Instrument logs for gas chromatography/mass spectrometry (GC/MS) volatiles shall reference the port used for each run where multiple ports exist.

e) Laboratories performing radiochemical analysis of Sandia samples shall record the data file names and dates for all calibration activities in the associated instrument logs. Procedures shall also require that the gas flow proportional counter (GFPC), alpha spectrometry, gamma spectroscopy, or alpha scintillation detector used to count each sample be logged.

2.7.2 Balances, volumetric pipettes, and sample storage refrigerators

a) Chemical and geotechnical analysis laboratories shall have a calibration SOP for analytical balances. The SOP shall specify that balances be checked daily (on all business days) against certified standards and that balances not accurate to within at least ± 1 percent be recalibrated or removed from service. The laboratory shall maintain logbooks in which the daily analytical balance calibration checks are recorded.

b) Chemical analysis laboratories shall have an SOP that requires daily temperature monitoring (on all business days) for refrigerated sample storage areas and the corrective action that will be initiated if a measurement falls outside the required range. The laboratory shall maintain logbooks for sample storage refrigeration units in which the daily temperature checks are recorded.

c) Chemical analysis laboratories shall have a calibration SOP for volumetric pipettes, other than glass pipettes, that deliver 100 microliters or more. This SOP shall specify that (1) pipettes be checked daily (on all business days) by weighing deionized (DI) water, and (2) pipettes failing to deliver to within ± 1 percent accuracy be recalibrated or removed from service.

2.7.3 Reagent water production

a) Chemical analysis laboratories shall have an SOP for reagent water or DI water production and system maintenance. This SOP shall outline specific control criteria for reagent or DI water quality, require daily water quality measurement (on all business days), and give specific corrective actions to be taken for out-of-control events.

b) Daily records of water quality shall be kept in logbooks designated for that purpose.

2.7.4 Control of standards

Chemical analysis laboratories shall have an SOP outlining policy on shelf life, labeling, and stock maintenance for reagents, stock solutions, intermediate dilutions, and working standards. Laboratories shall maintain standards preparation logs and standard certificates of analysis in an orderly manner to facilitate retrieval.

a) The SOP shall specify a shelf life no greater than one year for stock solutions prepared in the laboratory from salts or metals.

b) The SOP shall specify a shelf life of no greater than one year for intermediate dilutions and vendor-supplied stock solutions, other than radionuclide solutions, when the constituent concentrations are 1 milligram per liter (mg/L) or higher. General inorganic analyte solutions with constituent concentrations less than one mg/L shall be defined as working standards.

The one-year shelf life shall not apply to neat materials or unopened ampoules containing solutions of organic compounds. The manufacturer’s expiration date, if any, shall apply to neat materials and unopened ampoules containing organic standard solutions.

c) The SOP shall limit the shelf lives of opened ampoules and intermediate dilutions containing organic standard solutions to no greater than those given below. Shorter shelf lives given in the EPA methods shall supersede the specified guidelines.

Analyte	Shelf Life
Volatiles	7 days for gases
	180 days for non-gases
Total petroleum hydrocarbons	180 days for purgeable (gasoline range organics [GRO])
	365 days for extractable (diesel range organics [DRO])
Pesticides/PCBs/ herbicides	180 days
Semivolatiles	365 days
High explosives (HE)	365 days, ≥ 1,000 ppm, stock 30 days, all intermediate dilutions Daily prep, all working standards

d) The SOP shall specify that working standards for volatiles and general inorganic analyses, other than multi-element radial viewing inductively coupled plasma-atomic emission spectroscopy (ICP-AES) working standards, be prepared fresh daily. The SOP shall require that ICP-MS and axial viewing ICP-AES working standards having concentrations less than one part per million (ppm) be prepared fresh daily.

e) The SOP shall specify that multi-element radial viewing ICP-AES working standards be prepared fresh at least once a month.

f) The SOP shall specify that anion and nutrient stock solutions be kept in refrigerated storage. Refrigerated storage for standards is subject to the requirements of Section 2.7.2 (b) of this SOW.

g) For laboratories doing radiochemistry, the SOP shall limit radionuclide solution shelf lives to a maximum of five years or five half-lives, whichever is less. The SOP may allow verification of expired standards against National Institute of Standards and Technology (NIST)-traceable standards or require that they be discarded. Re-verification is allowed if the following criteria are met.

  i.) At least three verification measurements of a standard shall be used to determine the mean value and standard deviation of the verification results.

  ii.) The two-sigma (2σ) value defining the 95 percent confidence interval shall not exceed 10 percent of the mean value of the three verification measurements.

  iii.) The certificate value (NOT including any uncertainty) shall lie within the 95 percent confidence interval determined from the mean and two standard deviations of the three measurements. However, if the interval defined is narrower than the mean ± 5 percent of the mean, a ± 5 percent acceptance criterion may be used.

  iv.) The methodology used, performance requirements, and documentation practices must be discussed in the SOP.

h) The SOP shall require that stock solutions and intermediate dilutions prepared in the laboratory be logged in a standards preparation log. The SOP should give specific guidelines on what information is to be included in log entries. Expiration dates for solutions prepared from multiple sources shall coincide with the earliest expiration date of the starting materials.

i) Minimum labeling requirements for stock solutions and intermediate dilutions that are intended for long-term use shall be addressed in the SOP and should include the information listed below.

- Preparer's initials.

- Date of preparation.
- Matrix.
- Concentration of constituents, unless too many are contained to be listed on the label.
- Expiration date.
- Unique standard name that is traceable to a standards preparation log.

j) The SOP shall require that organic analysis calibration standards be prepared using high purity solvents that were accompanied by manufacturers' certificates of analysis when purchased.

k) The SOP shall require that standards for atomic spectroscopy be prepared in ASTM Type I water. The applicable ASTM standard for Type I water is the older standard that specifies a 16.67 mega ohm per centimeter (MΩ·cm) resistivity control criterion. Preparation water need not meet the newer 18.0 MΩ·cm criterion.

l) The SOP shall require that standards for radiochemistry and wet chemistry be prepared using ASTM Type II water, at a minimum.

m) The SOP shall specify that expired standards be segregated and labeled as expired while awaiting disposal.

n) Vendor-supplied solutions that are used as primary calibrants shall be NIST-traceable where possible.

2.7.5 Glassware

a) The laboratory shall have an SOP for glassware cleaning.

b) All volumetric glassware used to make standard and sample dilutions in SDR work shall be ASTM Class A glassware. Dilutions may also be accomplished by automation or by using pipettes and/or balances that are controlled in accordance with the applicable provisions of this SOW.

2.7.6 Incident tracking

Laboratories shall have a system for recording and tracking incidents involving breakage of reagents and client samples. This system is needed to help explain unexpected "hits" in samples that were analyzed during periods when the ambient air may have been contaminated. The tracking system may be implemented through facilities, health and safety, QA, or other laboratory groups.

2.8 Analytical and Quality Assurance Standard Operating Procedures

2.8.1 Control of Standard Operating Procedures

The laboratory shall maintain controlled copies of approved SOPs for each analytical method or general procedure performed by laboratory personnel. The laboratory shall set and demonstrably adhere to a schedule of periodic review for SOPs. Changes in laboratory SOPs that significantly affect the analysis or documentation of Sandia samples shall be transmitted to the SDR for approval prior to implementation. Laboratories may seek approval by telephone for minor SOP modifications. Geotechnical laboratories may use the most recent ASTM methods instead of SOPs, provided that there are no deviations from the method in practice.

2.8.2 Availability of Standard Operating Procedures

Controlled copies of SOPs shall be readily available to all personnel performing analytical work in support of the SNL/SMO. This may be accomplished either by issuing a copy to each analyst, electronically posting SOPs, or making a library of SOPs accessible to analysts. Policies for notifying laboratory staff members of SOP updates shall be defined.

2.8.3 Analyst familiarity with Standard Operating Procedures

Analyst familiarity with SOPs shall be documented to ensure that the contents of QA and analytical SOPs are effectively communicated to personnel performing analysis of Sandia samples. Laboratory procedures shall require that method training and QA indoctrination be performed and documented in training files.

2.9 Sample receipt and storage requirements

2.9.1 Chain of Custody forms

Sandia samples received by the laboratory will be accompanied by an Analysis Request and Chain of Custody (ARCOC) form. Sample custody will be transferred to the laboratory at the time of sample receipt, after which the laboratory will be responsible for maintaining unbroken COC. By definition, a sample is in custody if it is (1) in one's possession, (2) in view, or (3) in a controlled access area. The SNL ARCOC form is provided as Attachment 7.

a) At the time of sample receipt, this form will have been partially completed by the sampling team and should indicate the ARCOC number, contract laboratory name and contact, specific project information, sample indentification (ID), sample location detail, sample matrix, bottle type and volume, sample type, chemical preservative if needed, collection dates and times, date shipped, and method of shipment.

b) Individual sample bottles are labeled with the sample ID, sampling date and time, preservation method, sampler's identity, and comments.

c) The laboratory sample custodian receiving the samples shall verify that the information listed on the ARCOC form is correct and accurately describes the contents of the shipment.

2.9.2 Acknowledgment of sample receipt

At the time of receipt, the laboratory sample custodian shall sign and date the ARCOC form in indelible ink to acknowledge sample receipt and accept custody. The sample custodian shall note discrepancies between the samples listed on the ARCOC and those actually received on the ARCOC form and sample login worksheets.

Note: The laboratory shall include all airbills in the case file where possible and shall record all freight-carrier tracking numbers on the login records when the airbills cannot be removed intact. (See Sections 4.1.1 through 4.1.3 of this SOW for reporting requirements.)

2.9.3 Documentation of anomalies

The laboratory sample custodian shall note on the ARCOC form and sample login worksheets any irregularities observed with the shipment, temperature, preservation, condition, or custody seals of samples received. Login worksheets shall specifically identify any samples affected by such irregularities.

a) The pH of all aqueous sample fractions, preserved and unpreserved, shall be checked during sample login. (Exceptions to the login pH check requirement are Rn-222, tritium, iodine, volatile organic compound [VOC], total organic halides [TOX], oil and grease, total organic carbon [TOC], and urine samples. The pH of samples submitted for the exception analyses listed here shall be checked at the time of analysis.)

b) The allowable temperature for samples requiring cooling for preservation is ≤6°C. The actual temperature of sample shipments shall be noted on login worksheets.

c) If no anomalies are encountered for a sample shipment, a brief statement of that fact shall be provided on login worksheets and in the case narrative.

d) If samples requiring preservation with nitric acid arrive unpreserved or inadequately preserved, the laboratory must contact the SDR for instruction regarding whether to proceed with the analysis. If the laboratory is instructed to adjust the sample pH, metals samples must be held for 16 hours and radionuclide samples must be held for 24 hours prior to withdrawing an aliquot for analysis.

2.9.4 Communication of anomalies

A laboratory representative shall notify the SDR immediately by telephone of any irregularities noted during the sample receiving process. In addition, the laboratory shall notify the SDR immediately if a sample shipment does not include sufficient sample volumes to meet the QC requirements of this SOW. Any problems with a sample shipment that adversely affect data quality shall be described in the case narrative that accompanies the report of analytical results for that delivery order.

2.9.5 Sample retention

The laboratory shall retain and store all Sandia samples associated with a specific delivery order for a period of 65 days after issuing the analytical report for that delivery order. The samples should be stored in such a manner that the analyses could be repeated or new analyses requested. Unused samples shall be stored in controlled and secured environments for 30 days, the lab will store the samples for an additional 35 days by whatever means is economical. ARCOC sample groupings shall be maintained.

2.9.6 Sample disposal

The laboratory is solely responsible for lawful disposal of all SDR samples after the 65-day sample storage requirement is fulfilled if the exception given in item (a) below does not apply.

a) If, due to the nature of the samples, the laboratory has no outlet for disposal or disposal is prohibitively expensive, then samples may be returned to the SNL/SMO.

b) If samples are to be returned to the SNL/SMO, the laboratory shall provide notification that includes an inventory of samples, ARCOC numbers, and radiation survey results to the SDR at least two weeks prior to shipping. Samples shall be packaged for return to SNL/SMO by ARCOC groupings and shall be returned within 95 days of report delivery at the SNL/SMO. Samples shall be returned under the exclusion provision in the Code of Federal Regulations 40 CFR 261.4. Samples shall be packaged and returned in the same manner as they were received. Samples can be returned by the most economical means available at laboratory expense. The laboratory is solely responsible for the lawful shipping of samples back to the SNL/SMO.

c) Laboratories shall not return extracts or digestates to the SNL/SMO.

d) Laboratories shall follow sample return instructions as indicated on the ARCOC when an expedited return is requested. This exception supersedes both Sections 2.9.5 and 2.9.6(b), above.

2.9.7 Return of shipping coolers and bottle orders

Laboratories shall initiate the shipment of sample coolers and blue ice back to the SDR within five days of receipt. Sample coolers can be shipped by most economical means at laboratory expense to the address shown below unless different instructions are provided by the SNL/SMO. This address shall also be used for the shipment of bottle orders.

Sandia National Laboratories
Sample Management Office
Building 928
1515 Eubank SE
Albuquerque, NM 87123

2.9.8 Laboratory-supplied sample containers

The SDR requires the laboratory to supply sample containers for most projects. The schedule of prices provided in the laboratory's proposal shall include discount prices for projects not requiring the laboratory to provide sampling containers. The SDR will initiate most sampling projects with a bottle order submitted to the laboratory. The laboratory shall provide pre-cleaned sample containers with the appropriate preservative for each analysis covered in the order. Container and preservative requirements are provided in Attachment 5 of this SOW.

2.10 Holding time requirements

2.10.1 Holding times

Analytical holding times are specified in Attachment 5.

2.10.2 Definition

Holding times are calculated in days or hours, according to the time units used in the EPA holding time requirements. If the EPA-specified holding time is given in hours, then the analysis must be complete before the end of the last hour of the holding time when calculated from the sampling time. When the holding time is given in days, the analysis must be complete before the end of the day on which the holding time would expire as calculated from the sampling day.

Holding time to extraction and holding time to analysis specifications given in EPA guidance and Attachment 5 shall be observed. Laboratories shall not meet holding times to extraction by initiating and then halting extraction procedures simply to avoid expiration of the holding time. That is, once begun, extraction procedures must be carried through. Methods that specify holding times to analysis but do not specify holding times to extraction shall have the analysis started by the expiration of the holding time. That is, samples may not be extracted and then held for later analysis simply to avoid expiration of the holding time.

2.10.3 Matrix types

Where matrix-specific holding times are not given in Attachment 5, the specified holding times shall apply to all sample matrix types.

2.10.4 Meeting holding times

It is crucial that the laboratory perform chemical analyses within the specified holding times. The laboratory shall promptly notify the SDR if it determines, upon sample receipt or thereafter, that one or more analyses cannot be performed within the holding time(s). Analyses not performed within the holding time and reported without prior explanation and SDR approval will not be paid for.

a) The SDR will make every effort to notify the laboratory when samples having less than 72 hours of the holding time remaining are to be shipped.

b) For samples with holding times greater than 48 hours, shipments arriving at the laboratory with less than 72 hours of the holding time remaining may be invoiced at the appropriate accelerated turnaround premium price. That is, when less than 72 hours remain, the 72 hour negotiated turnaround price will apply.

2.10.5 Violations

The laboratory shall provide an explanation for all holding time violations in the case narrative. Laboratories shall not allow sample analyses to be canceled because the holding times could or will be missed without prior notification of the SNL/SMO. Laboratories that repeatedly cancel analyses without notifying the SDR may be suspended from the Sandia laboratory analysis program or Sandia may cancel the contract for cause in accordance with the clause titled, “Cancellation or Termination for Convenience” (SF-6432-CS [02/01/12]).

2.11 Laboratory data verification and review requirements

2.11.1 Worksheet review

a) All analyst worksheets describing analysis of Sandia samples shall undergo supervisory or peer review. A field shall be provided on each worksheet for the reviewer's initials. The reviewer need not sign each page of a submittal; only one signature per data submittal (per analytical batch) is required.

b) Worksheet review signatures signify that the analyst has met the requirements of the method, laboratory QA policies, and this SOW.

2.11.2 Report review

All data transmitted to the SDR by the laboratory shall undergo data verification and completeness review by the laboratory's QA or technical staff. In addition, reviews shall include 100 percent verification of agreement between EDDs and hard copy reports, as defined in Section 2.2.1(s) of this SOW, until the efficacy of the EDD production process is demonstrated. Signature evidence of these reviews in the case narrative is required.

2.12 Laboratory record maintenance requirements

The laboratory shall maintain a case file containing all documents and records associated with each specific delivery order for the duration of the contract period. Alternatively, an effective system ensuring the ability to retrieve all associated records in a timely fashion may be implemented. All raw data, worksheets, run logs, digestion logs, shipping and login records, custody forms, and communication records must be included in the case file or addressed by the retrieval system discussed above. This supporting documentation may be used to verify compliance with the requirements outlined in this document or to support the data in a court of law. The supporting documentation shall be shipped to the SDR or discarded, at the discretion of the SNL/SMO, when the contract base period and all exercised extensions expire. Charges for shipping supporting documentation will be reimbursable at cost.

If an electronic data storage system is used, the laboratory shall have an SOP that addresses creating, verifying, and tracking electronic records. The Good Automated Laboratory Practices (GALP) requirements of Section 2.17 of this SOW shall be implemented as applicable, and the records shall be in a format that is readable using common commercial software.

2.13 Corrective action for out-of-control events

2.13.1 Requests for Corrective Action Reports

The laboratory may be required to provide a CAR for any out-of-control event associated with analytical services provided to Sandia.

2.13.2 Delivery of Corrective Action Reports

As described in Section 2.3.3 of this SOW, initial responses to CAR requests, including the projected schedule for completion, shall be due no later than two weeks from the date of the request. The SDR reserves the right to request delivery of CAR responses in less than two weeks if circumstances indicate that this is necessary. Repeated failure to submit requested CARs may result in suspension of the laboratory from the Sandia laboratory analysis program or Sandia may cancel the contract for cause in accordance with the clause titled, “Cancellation or Termination for Convenience” (SF-6432-CS (02/01/12)).

2.14 Quarterly progress report requirement

2.14.1 Contents of Quality Progress Reports

The laboratory shall submit QPRs to the SNL/SMO. QPRs shall address calendar quarters and are due by the 15th day of the month following the reporting period. In addition to the quarterly reporting requirement, laboratories will notify the SDR immediately for issues relating to items (d), (e), and (f) below. Emphasis should be placed on the following:

a) List new analytical methods implemented and/or any substantive changes to existing methods. This should also include changes in capabilities due to acquisition or retirement of equipment.

b) Summarize all out-of-control incidents that occurred during the reporting period. Also, include a summary of non-conformances identified during external and internal audits. Non-conformance reports issued as a result of QC or reporting failures need not be summarized unless they involved chronic problems that necessitated formal corrective action plans.

c) Report significant changes to the LQAP, paying particular attention to those that affect the analysis or documentation for Sandia samples. If new MDLs or QC acceptance criteria were established during this period, report the new MDLs and/or acceptance criteria in QPRs. Reports on new MDL studies should include a summary of the method blank evaluation (Section 3.3.1 (c) and (d) of this SOW).

d) Discuss any management, QA personnel, key technical personnel, or supervisory changes. Any significant reduction in force or backup coverage for key positions should also be reported.

e) Report any changes in accreditation or certification, including voluntary withdrawal from any certifying program. Utah certification status and/or Nuclear Regulatory Commission license agreements are extremely important.

f) Provide all PT results that were graded during the reporting period, and include any corrective action plans initiated in response to PT failures. PT results should be reported in Microsoft Excel electronic format.

If no significant changes occurred during the reporting period, and if no CARs were generated, then a simple statement of these facts shall suffice to meet the QPR requirement.

2.14.2 Compliance

Repeated failure to comply with the QPR requirement in this SOW may result in suspension of the laboratory from the Sandia laboratory analysis program or Sandia may cancel the contract for cause in accordance with the clause titled, “Cancellation or Termination for Convenience” (SF-6432-CS (02/01/12)).

2.15 Primary contact person

2.15.1 Laboratory contact person

The laboratory shall assign a project manager to be the primary contact person for issues relating to the analysis of Sandia samples.

2.15.2 Sandia Delegated Representative contact person(s)

The technical representatives shall be the SDR(s) named in the contract.

2.15.3 Communication

Open communication between the SDR and laboratory is critical in the development of a mutually satisfactory business relationship. Laboratory technical representatives are encouraged to seek guidance in advance of performing work when any questions arise and to comment on any analytical approach they may believe to be flawed.

2.16 Radioactive materials license requirements

All participating laboratories shall have a current radioactive materials license that is appropriate to the materials they anticipate receiving under this contract. If the radioactive materials license has expired, the laboratory shall have a letter of timely renewal on file. Photocopies of new or updated licenses shall be provided to the SDR with the next QPR.

2.17 Good automated laboratory practices

The degree of dependence upon automated calculation routines and information stored in modern laboratory databases indicates a need to ensure the integrity of software and information. GALP must be used by the laboratories to ensure the reliability of data. These include traceability, accountability, standardized procedures, adequate resources, and the availability of documentation of conformance to the requirements (including setting acceptance criteria where appropriate). Chemical analysis laboratories and, to the extent applicable, laboratories performing asbestos and geotechnical tests must have procedures that address the issues listed below.

2.17.1 Laboratory management

When electronic data are collected, analyzed, processed, or maintained, the laboratory management shall:

a) Ensure that personnel clearly understand the functions they are to perform.

b) Ensure that QA staff members monitor computer activities.

c) Ensure that personnel, resources, and facilities enable work and are available.

d) Receive reports of audits of Laboratory Information Management System (LIMS) and other computer systems and ensure that corrective actions are promptly taken in response to any deficiencies.

e) Approve the SOPs setting forth the methods that ensure electronic data integrity; ensure that any deviations from SOPs and applicable GALP provisions are appropriately documented and that corrective actions are taken and documented; and approve subsequent changes to SOPs.

2.17.2 Personnel

The laboratory shall ensure that all computer support staff and users:

a) Are trained with experience to perform assigned functions.

b) Have a current summary of their training, experience, and job description, including their knowledge relevant to LIMS design and operation, maintained at the facility.

2.17.3 Quality assurance

The laboratory shall designate QA staff to monitor computer functions and procedures. QA staff members shall:

a) Be entirely separate from and independent of LIMS personnel and shall report directly to management.

b) Have immediate access to LIMS data, SOPs, and other records pertaining to the operation and maintenance of LIMS.

c) Audit the computer systems to ensure the integrity of the electronic data and prepare audit reports. Reports shall describe the operation audited, the dates of the audit, the person performing the audit, findings and problems observed, action recommended and taken to resolve existing problems, and any scheduled dates for re-audit. QA staff shall report to laboratory management any problems that may affect data integrity.

d) Determine that no deviations from approved SOPs were made without proper authorization and adequate documentation.

e) Ensure that the responsibilities and procedures applicable to QA, the records kept by QA, and the method of indexing such records are properly documented and maintained.

f) Establish nonconformance and corrective action procedures for hardware and software failures.

2.17.4 Electronic data

Electronic data shall be managed in such a way as to ensure and/or include:

a) Electronic data storage media are identified and indexed. These processes shall be included in laboratory SOPs.

b) The individuals responsible for entering and recording data are uniquely identified when the data are recorded, and the times and dates of entry are documented.

c) The instrument transmitting electronic data is uniquely identified when the data are recorded, and the time and date of transfer are documented.

d) Procedures and practices for verifying the accuracy of data are documented and included in laboratory SOPs.

e) Procedures and practices for making changes to electronic data are documented and provide evidence of change. Such evidence should preserve the original data, include the date of the change, indicate the reason for the change, identify the person who made the change, and, if different, identify the person who authorized the change. These procedures shall be included in laboratory SOPs.

f) Procedures and practices for backing up electronic files are documented. These procedures shall include frequency, storage, and the process for restoring files. These procedures shall be included in the laboratory SOPs.

2.17.5 Software

Software shall be managed in such a way as to ensure and/or include:

a) Approved Standard Operating Procedures exist for:

   i. Verification and validation procedures to verify that all software programs accurately perform the intended functions. These procedures should address software security (cell protection, for example). When indicated, change control procedures shall include reporting and evaluating problems and implementing corrective actions.

   ii. Version control procedures that document the software version currently used and its implementation date.

   iii. Maintaining a historical file of software including dates of use, software operating procedures (manuals), software changes, and software version numbers.

b) Laboratory management shall ensure documentation for the issues in item (a) above is maintained and is readily available in the facility where the software is used.

2.17.6 Security

Laboratory management shall make sure security practices are in place to ensure the integrity of electronic data that:

a) Ensure calculation routines are secure from inadvertent changes.

b) Require a login password to access stored data, enter new data, and change existing data.

c) Establish access categories (read only, read/write, read/write/change) as appropriate to the duties of staff members.

2.17.7 Hardware

Laboratory management shall ensure that hardware and communications components are:

a) Well-documented with a detailed description including design and capacity.

b) Installed and operated in accordance with manufacturer's recommendations and, at installation, undergo acceptance testing. SOPs shall be established and maintained to define the acceptance criteria, testing, documentation, and approval requirements for changes to LIMS hardware and communication components.

c) Inspected and maintained on an ongoing basis. Non-routine maintenance shall be documented, including a description of the problem, the corrective action, and the acceptance testing performed to ensure that the hardware or communications components have been properly repaired.

2.17.8 Comprehensive testing

Laboratory management shall ensure that comprehensive testing of LIMS performance is conducted, at least once every 24 months or more frequently as a result of software (Section 2.17.5) or hardware (Section 2.17.7) changes or modifications. Testing will be documented and shall be retained and available for inspection or audit.

2.17.9 Records retention

Laboratory management shall ensure that SOPs for records retention are implemented and that staff members follow the SOP specifications.

2.17.10 Facilities

With regard to facilities, laboratory management shall ensure that:

a) The environmental conditions of the facility housing the hardware are appropriately regulated to protect against data loss.

b) Environmentally controlled storage capacity is provided for retention of electronic data, storage media, and records pertaining to the computer systems.

2.17.11 Standard operating procedures

Laboratory management shall ensure that:

a) Each current SOP is readily available where the procedure is performed.

b) SOPs are periodically reviewed and reviews are documented to ensure they accurately describe the current procedures.

c) SOPs are approved and changed in accordance with laboratory QA policy.

d) A historical file of SOPs is maintained.

2.18 Records for method development and initial demonstration of proficiency

The LQAP or procedures must specify the records needed to document method development and initial demonstration of proficiency. A system for tracking and retrieving these records must be in place.

3.0 ANALYTICAL AND QUALITY CONTROL REQUIREMENTS

The QC requirements specified in this SOW represent the minimum requirements for SDR work. However, if a requested method includes QC requirements that are more restrictive than those specified in this SOW, then the more restrictive QC requirements must also be met.

3.1 Standard preparation and instrument calibration requirements

3.1.1 Working standards

Standard preparation for analysis of Sandia samples shall be performed according to the specifications in items (a) through (n) of Section 2.7.4 of this SOW. Working standard preparation information shall accompany daily analysis worksheets and shall be sufficiently detailed to demonstrate compliance. Initial calibration verification (ICV) solutions, if applicable, shall be documented in sufficient detail to make clear they were derived from a source different from that used to prepare the calibration standards.

3.1.2 Calibration

Instrument calibration shall be performed according to the specifications of the SW-846, ASTM, or other method where applicable. Calibration for analytical techniques that are not addressed in industry-standard methods shall be performed according to the specifications of the analytical procedure adaptations used by the laboratory. Minimum calibration requirements specific to this SOW are given below. The requirements in item (a) apply only when instruments are in use.

a) Instruments used to acquire general inorganic data shall be calibrated daily or once every 24 hours, and each time the instrument is set up.

  i. In conformance with SW-846 Update IV Method 6010C, ICP-AES calibrations shall consist of a blank and at least three standards. Exceptions can be made for analytes that commonly have comparatively high concentrations in environmental samples, such as Na, Ca, Mg, Fe, and Al. For trace metals analysis by axial-viewing ICP-AES, and particularly for As, Cd, Pb, Sb, Se, and Tl, suggested standard concentrations are a blank, one standard in the 50- to 100-parts per billion (ppb) range, one standard in the 250- to 500-ppb range, and a standard not exceeding 1 ppm. Laboratories may include an additional standard at a higher concentration if desired to extend the calibration range.

ii. The four-point calibration requirement specified immediately above should be applied as the minimum allowable for all general inorganic instrument calibration curves that are derived using linear regression. Where second-order polynomial regression is used, the minimum requirement is a blank and five standards. In all cases, laboratories must adhere to more restrictive requirements found in individual methods. No polynomials of order higher than two shall be used in any analysis.

iii. When inorganic anions are analyzed by ion chromatography (IC), laboratories may choose one of two approaches. Daily calibration is recommended. However, laboratories may continue to use existing calibrations provided that appropriate control criteria are met. In the latter case, continuing calibration verification (CCV) and continuing calibration blank (CCB) analyses must be conducted at the beginning of each analytical run and must meet the standard acceptance criteria specified in this SOW. In addition, the ± 10 percent retention time control limits specified in Method 9056 (Section 7.1.4) and Method 300.0 (Section 9.4) shall be used. However, laboratories shall adhere to the additional requirement that the maximum deviation for the CCV analyte peak centroids from the average retention times obtained during calibration shall be 0.5 minutes.

b) Instruments used to acquire radiochemical data shall be calibrated at the frequency specified in Section 3.6.9 of this SOW.

c) Instruments used to acquire organic data shall be calibrated at the frequency specified in the applicable EPA method or this SOW. Conflict between EPA methods (for example, SW-846 vs. 600 series methods) exists in some cases. When organic chemistry calibration requirements are given in this SOW, those requirements are the minimum allowable. When calibration criteria are not specified either in the method or in this SOW, calibration curves will be generated using at least five standards and shall have a correlation coefficient of at least 0.99. Calibration curves shall not be forced through zero. Calibration requirements for instruments used in routine organic chemistry are discussed in detail in Section 3.5.2 of this SOW.

d) Instruments used to acquire inorganic and organic data that utilize linear or quadratic curves shall have initial calibration Y-intercept absolute values ≤3 times the MDL. If the magnitude of the concentration-axis intercept is equal to or greater than 3 times the MDL, the laboratory must (1) recalibrate, or (2) raise the MDL and Practical Quantitation Limit (PQL) by a sufficient margin to adequately address the deficiency, or (3) for organic chemistry only, apply a weighted linear calibration approach.

3.2 Sample analysis requirements

3.2.1 Worksheet requirements

Analyst worksheets used to record analytical data shall present a complete record of all information pertinent to the analysis. Analyst worksheets may be computer-generated or hand-written using indelible ink. A completed analyst worksheet that includes the information listed below is required for each analytical run.

a) The name of the person who performed the analysis.

b) The instrument used in the analysis. If the laboratory has more than one instrument of a particular model, a unique designation shall be given to each.

c) The name or initials of the peer, supervisory, or QA reviewer. (See Section 2.11.1 of this SOW for specific review requirements.)

d) Calibration information as specified in Section 2.2.1(h) of this SOW. Radiochemistry counting instrument calibration information should be limited to calibration dates, computer data file names, and a statement certifying that calibrations were successfully performed on schedule.

e) Standards information, including the name, preparation date, and expiration date of calibration and calibration verification standards, as applicable.

f) The analytical procedure and regulatory method used.

g) The equations for calculations used to obtain results. If instrument readouts give results, without the need for further mathematical manipulation, the worksheets shall include the statement “result = instrument readout.”

h) The date and time that the analysis was performed.

3.2.2 Sample preparation

Sample preparation shall be conducted according to the specifications of the analytical procedures, except as noted below.

a) Samples shall be digested/extracted according to the procedures given (or referenced) in the appropriate EPA method unless different procedures are specified in the analysis request (see Section 2.1.1(b)). Unless specifically asked to do otherwise by the SNL/SMO, laboratories may use sonication rather than Soxhlet and separatory funnel rather than continuous liquid-liquid extraction as applicable for organic extractions. Accelerated solvent extraction (ASE) is not allowed unless rigorous method development data are submitted and approved in advance.

b) Laboratories working under general inorganic contracts shall specify prices for EPA 200.2 digestions, SW-846 digestions, total dissolution digestions (HNO_3, $HClO_4$, HF), toxicity characteristic leaching procedure (TCLP) extractions, and synthetic precipitation leaching procedure (SPLP) extractions. All metals digestions and sample dilutions shall be performed using ASTM Type I water.

Unless the SDR gives specific exemption, the 3050 digestion for soils that uses HCl (digestion for ICP-AES 6010) shall be used for Sn, Sb, and Ag. These analytes recover very poorly when HCl is not used, as in the 3050 digestion for Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) 6020. Since this is true, the 6020 digestion MDLs for these metals, as determined using aqueous spikes, are probably unreliable. If laboratories wish to use a dilution of the 6010 digestate and run by ICP-MS 6020, MDLs must be determined in advance using the exact procedure proposed.

c) Soil samples submitted for radiochemical parameter analysis by techniques other than gamma spectroscopy shall be dried, crushed to -200 mesh, and homogenized prior to analysis. Gamma spectroscopy samples shall be dried, crushed to -28 mesh, and homogenized prior to analysis. Tritium samples are exempt from this sample preparation requirement.

i. The entire sample shall be crushed and homogenized, up to a maximum of 200 grams, unless a portion is needed for another analysis that does not require this preparation. Timed grinding may be used in lieu of sieving if the contractor develops and maintains method-development data proving efficacy.

ii. Extraneous material that cannot be crushed (such as metal debris and organic matter) may be removed from samples.

iii. Solid samples that are submitted for radiochemical analysis (other than gamma spectroscopy) shall be subjected to a total dissolution digestion.

iv. The SDR will specify the sample preparation techniques for radionuclide determinations by general inorganic analytical techniques. For example, ICP-MS uranium isotopic ratio data will typically be acquired using a 3050 digestion for soils and a 3010 digestion for waters. However, uranium bound in the silicates in soil samples will not be measured if a 3050 digestion is used.

This means that the total uranium values obtained by ICP-MS from a 3050 digestate can be biased low. If accurate total uranium data are required, the SDR may request that a total digestion be used. If a total digestion is required, SW-846 method 3052 may be an option. That method does employ hydrofluoric acid (HF), but uses boric acid to consume the HF to protect instrumentation.

d) Percent moisture measurements shall be made and reported for all Sandia soil samples submitted for analyses, including tritium. Unless otherwise specified, soil sample results for all analyses shall be reported on an as-received basis.

e) Percent moisture data does not need to be included with geotechnical test results.

f) Extraction procedures for soil samples submitted for anion analysis will be selected on a case-by-case basis. The PQLs presented in Attachment 1 assume a nominal 1:20 dilution factor.

g) Percent moisture data shall be acquired for all biota samples. For metals and radionuclides, biota samples may be freeze-dried, blended, and digested for analysis. An ashing step may be added for nonvolatile radionuclides. For organic chemistry analyses (other than VOCs), the procedure described below is suggested. The procedure below may also be used for metals and radionuclides if desired.

  i. Cut the samples into small pieces with clean, stainless steel tools (knife or scissors).

  ii. Place the sample pieces into a clean, ceramic mortar, freeze them with liquid nitrogen (LN_2), and grind the frozen pieces with a clean, ceramic pestle. Add LN_2 as necessary to keep the sample frozen.

  iii. Take aliquots of the samples by allowing them to partially thaw, mixing them for further homogenization, and then weighing them into the digestion/extraction vessels. Another aliquot of this homogeneous mixture should then be weighed and taken to complete dryness for percent moisture determination.

  iv. Water loss by sublimation during the grinding step may be accounted for by weighing the sample before and after grinding. This water loss should be combined with that from step (iii) above to calculate the total percent moisture for the sample.

  v. Biota samples shall be reported as fresh weight for all Sandia samples.

h) Digestion and processing requirements for swipes, filters, and bioassay samples will be communicated to the laboratory prior to sample shipment. Special requirements for other sample matrices will be handled on a case-by-case basis.

i) The laboratory shall develop a procedure for sub-sampling and obtaining a representative aliquot. In addition, the laboratory shall be prepared to incorporate the sub-sampling procedure specified in Attachment 10, Routine Sub-sampling Procedure.

3.2.3 Initial dilution of samples

Since some Sandia samples have a high solids content, initial dilution of samples for analysis by graphite furnace atomic absorption (GFAA), ICP-AES, and ICP-MS will be allowed according to the criterion specified in item (a) below. Samples submitted for organic analyses may be initially diluted according to item (b) below.

a) Water samples having total dissolved solids (TDS) content greater than 2000 mg/L may be diluted, prior to analysis, by the smallest reasonable dilution factor required to bring the solids content down to below 2000 mg/L. If TDS analysis was not requested by the SNL/SMO, a sample's specific conductance may be used to estimate TDS. For this purpose, the specific conductance of the unpreserved sample fraction in micro ohms per centimeter (μmho/cm), multiplied by the factor 0.7, shall be considered equal to the sample TDS in mg/L.

b) For organic compound determinations, screening of samples against calibrated instruments to determine whether initial dilution is indicated shall be allowed. VOC screening methods are discussed in Section 3.5.4 (a) of this SOW. If all target analytes in the diluted sample are non-detects and no other interference is obvious, the sample will be reanalyzed at a lower dilution.

c) Samples diluted according to the criteria specified in items (a) and (b) above shall be listed and discussed in the case narrative. The MDLs and PQLs reported for such samples shall be elevated accordingly, as discussed in Section 3.3.3(b) of this SOW.

3.2.4 Analytical techniques and Standard Operating Procedures

The laboratory shall employ approved analytical techniques and SOPs in the analysis of Sandia samples. If a nonstandard technique is required to achieve a specific sampling objective, the laboratory will be asked to provide a schedule of charges for the work on a case-by-case basis.

a) The laboratory shall perform routine sample analyses using the analytical techniques and methods specified in Attachments 1 through 4. Approved adaptations of EPA, APHA, ASTM, NIOSH, OSHA, or other methods that employ the specified analytical techniques shall be used. Adaptations of such methods to the specific laboratory environment shall refer to the parent procedures.

b) If, due to catastrophic instrument failure, the specified technique(s) cannot be used, a laboratory representative shall contact the SDR to obtain approval for the use of an alternate technique. If the proposed alternate technique will not yield results suitable to the end use of the data, instructions for shipment of samples or sample splits to another laboratory will be provided by the SNL/SMO.

c) In the event that samples or sample splits must be sent by the laboratory to another laboratory, the laboratory initiating the shipment shall be responsible for demonstrating unbroken COC up to the time of shipment and for ensuring that the samples are properly packed for shipment.

3.2.5 Chromatographic peak integration guidelines

The default integration technique that should be used for peaks that are not resolved to baseline is the drop technique, where the peak is integrated from horizontal baseline to the valley between the two peaks. This integration method results in the most accurate peak area determination for all situations, except those where two peaks are not fully resolved and there is a large difference in size between the two peaks. In these cases, large quantitative errors for the smaller peak may occur if an incorrect integration method is used. Specifically, when a large difference in relative peak size is encountered, and the smaller peak elutes after the larger peak, using an incorrect integration method will result in large positive quantitation errors[1].

When two peaks are not fully resolved and the smaller peak elutes after the larger peak, the smaller peak should be integrated using a tangential, exponential or gaussian skim technique, where the peak is integrated from the valley between the two peaks to the point where it rejoins the tailing baseline. A skim integration technique should only be used when the area ratio of the two peaks is 5% or less, based on the original integration using the drop integration technique.

3.3 Detection limits, reporting requirements, and QC exemptions

3.3.1 Method Detection Limit determination

The MDL for all organic and general inorganic parameters shall be determined, and verified (per Section 3.3.1(a)), and the results submitted to the SDR annually. Exceptions to this requirement are given in items Section 3.3.1 (a)(i) and (a)(iv) below. MDLs from the most recent MDL study shall appear on the Analysis Results forms.

a) The MDL is defined to be the point at which the observed signal can reliably be considered to be caused by the analyte being measured.

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[1] Bicking, M., Integration Errors in Chromatographic Analysis, Part II: Large Peak Size Ratios, *LCGC* [Online], **2006**, 24, 1.

Unless superseded by new EPA guidance, MDL determinations shall be performed as specified in 40 CFR 136 or the latest EPA acceptable procedure. Preparation of the standard solutions shall include all preparation steps (digestion, filtration, extraction, distillation, etc.) that would be used in the preparation of environmental samples.

i. As an alternative to annual MDL determinations, laboratories may perform the MDL studies once and then perform empirical verification of MDLs quarterly thereafter. If MDL studies are performed annually, empirical MDL verifications must always be performed subsequently to show adequate sensitivity at the new MDL concentration. Empirical verifications of existing MDLs may be done with a single standard prepared at the MDL concentration (in a standard volume of water, if applicable).

    1. The laboratory's procedure for empirical MDL verification should be added to the MDL determination SOP.

    2. For future empirical verifications, the existing MDLs should be set to zero as necessary to avoid truncating low-level data at the existing MDL levels.

    3. If MDLs are set to lower levels following new empirical verifications, the populations of method blanks should be monitored to ensure ambient contamination is not subsequently reported as positive analyte detections.

    4. Guidance for determining "what a detection is" should be included in the revised MDL SOP. The recommended standard is a signal-to-noise ratio of no less than 3:1 using the average noise signal.

    5. Guidance for documenting judgment calls should be included in the revised MDL SOP.

ii. The VOC water MDL can be used for the VOC low-level soil analysis MDL.

iii. Soil sample MDL determinations for organics may be performed with or without using muffled sand, an appropriate salt, or other soil matrix substitute. The specific choice of approach to soil substitutes is left to the laboratory's discretion.

iv. Due to the precision (good or bad) that is attainable for low level standards in certain organic methods, laboratories may believe that the MDL values obtained on any particular day are of little technical value. Laboratories may suggest modified values to be used upon the likelihood of producing false positive or false negative results. In any case, if false negative results are expected (see below), the MDL study should be repeated. The

SDR will determine whether the use of alternate values for MDLs is acceptable on a case by case basis.

The SDR has encountered incorrectly calculated MDLs many times, typically resulting from one of two types of problems (see below). In addition, comparison of the MDLs derived at various laboratories for the same analyte and method shows variations of up to two orders of magnitude. Obviously, this suggests strongly that an error exists in one or more of the MDL studies.

1. In the first case, standards having inappropriately high concentrations are used in the MDL studies, resulting in MDLs that are too high (the standard deviation of large numbers is a large number). This situation is discussed explicitly in 40 CFR 136, Appendix B, paragraph (4)(b) in the procedure section. This error can result in false negative results. 40 CFR 136 suggests an iterative approach to MDL determination to address this circumstance.

2. In the second case, the combination of extraction methods that tend to yield low recoveries with analytical techniques that yield good precision at low levels can result in calculated MDLs that are too low. That is, laboratories cannot actually “see” an analyte spiked into a standard volume of water at the level of the calculated MDL. This error can result in false positive results. 40 CFR 136 does not specifically address this circumstance.

In keeping with the intent of the 40 CFR 136 language, and to address the ubiquitous MDL problems encountered in Sandia work, laboratories are required to verify their calculated MDLs empirically. A suggested approach for verification of MDLs, neglecting the possible matrix effects of real field samples, is extracting standard volumes containing target analytes at approximately 0.5, 1, 2, and 5 times the calculated MDLs. In general, laboratories should use 5:1 as the target signal-to-noise ratio for MDL concentrations. Examination of the resulting data will indicate whether the calculated MDL should be artificially elevated or the MDL study should be repeated using different analyte concentrations.

A “detection” in MDL verification is defined as a result meeting all the qualitative identification criteria in the method (for example, as discussed in Section 7.6.1 of SW-846 method 8260C). Verify the MDL on each instrument by analyzing a reference matrix spiked at a concentration very near to the MDL concentration. If the analyte is not detected, repeat the test at a spiking level 2 times higher and raise the MDL accordingly.

v. In some cases, the available wavelengths in ICP-AES may consistently yield MDLs very close to, but higher than, the detection limits specified in Attachment 1. In such an event, the laboratory may be granted permission by the SDR to report to the slightly elevated MDLs.

vi. MDL studies are not required for acidity, alkalinity, biological oxygen demand (BOD), color, corrosivity, dissolved oxygen, gravimetric oil and grease, hardness, ignitability, pH, titrimetric sulfide, conductivity, any of the solids methods, or turbidity.

b) If any MDL result is greater than the corresponding Sandia target MDL, a discussion of the problem and planned corrective action shall accompany the report deliverable. This requirement is waived where an existing prior agreement allows slightly elevated MDLs for some parameters. Failure to implement effective corrective action may render the laboratory ineligible to receive samples for which determination of that parameter is requested.

c) The reports for MDLs should be in tabular summary form and included in the QPR (Section 2.14.1). Raw data generated in the determination of MDLs shall not be included as part of the deliverable, but may be specifically requested for examination by the SDR during audit and data package assessment activities.

d) Ambient low-level contamination and other problems (such as inconsistent baseline) can make it impossible to say whether a detection above the MDL truly represents analyte in the sample. In this case, large numbers of false positives can result and the definition of MDL is not met. Laboratories shall scrutinize method blank populations for all organic and general inorganic parameters whenever MDLs are updated, or annually at a minimum. Populations of method blank data must be examined to assess the ability to reliably detect analyte at the MDL without interference from instrument noise or ambient contamination. A summary of this study should be included in the MDL study file and in the QPR.

If method blank "hits" exceed 10 percent of the total population, a new test MDL shall be derived. Unless superseded by new EPA guidance, the laboratory shall set a new test MDL at $X + \sigma$, where X is the mean of the positive method blank hits and σ is the standard deviation of the positive method blank hits. The population of method blank results shall then be reevaluated against this new MDL. If the number of hits is reduced below 10 percent, then the test MDL should be implemented. Experience has shown this approach to be effective. However, the laboratory should contact the SDR if it fails to meet the objective of reducing false positives to single-digit values.

3.3.2 Practical quantitation limits

PQLs or reporting limits (RLs) shall be reported with all organic and general inorganic analysis results. PQLs shall be defined by the lowest concentration used in the calibration curve and should be a concentration that is approximately 5 to 10 times greater than the MDL for the associated parameter. If RLs are specified, then all PQL references shall imply the RL values. For the parameters exempted from MDL determinations in Section 3.3.1(a)(vi), the applicable laboratory PQLs shall accompany analytical results in place of the MDL.

3.3.3 Reporting conventions

a) General inorganic and organic chemistry results shall be accompanied by both the MDL and PQL.

b) MDLs and PQLs shall be adjusted to reflect the conditions for the specific sample. That is, the MDLs and PQLs shall reflect dilution factors and sample aliquot sizes used in the analysis of each sample. For reporting purposes, the PQLs and MDLs shall be reported to no more than two significant figures (see Section 4.1.13).

c) Organic and general inorganic results that are less than the MDL shall be reported as Not-Detected (ND) and qualified with a “U“ flag. Results between the MDL and the PQL shall be qualified with a “J“ flag as estimated.

d) Negative sample results with absolute values > the PQL shall require sample dilution and adjustment of the MDL and PQL as directed above in Section 3.3.3(b). This requirement is intended to address significant matrix-related signal suppression.

3.3.4 Radiochemistry detection limits

The SDR requires a means by which to capture sample-specific information, such as sample weight/volume, counting time, and chemical recovery that affects a laboratory’s ability to detect radiochemical analytes. The detection limit calculations in this section incorporate data that are specific to both the sample and the detector it is counted on.

The calculations given in items (a) and (b) below apply to detectors for which at least 35 background counts can be obtained. For low-background alpha spectrometry, it is very difficult to obtain a sufficient number of background counts to support the assumption of normal distribution. In that case, the assumptions underlying the equations in items (a) and (b) break down, resulting in an inappropriately large number of false positives. If at least 35 background counts cannot be obtained, laboratories shall use the low-background detection limit calculation of item (c). Also, should they wish to do so, laboratories may apply the detection limit calculation approach of item (c) to detectors having backgrounds above 35 counts.

a) Radiochemistry laboratories shall calculate a sample-specific (concentration corrected) Critical Level (L_c) for each radiochemical parameter. The L_c values shall be calculated according to the equation below and reported with each analytical result submitted to the SNL/SMO. A sample or blank will be considered to have activity above the applicable background only when the sample or blank concentration exceeds the L_c. This calculation gives the level at which there is a 5 percent probability of reporting a false positive result for a sample or blank containing no activity.

$$L_c = \frac{1.645(2*TBC)^{1/2}}{2.22DEIVTRA}$$

Where:

TBC = total background counts
2.22 = DPM/pCi
D = decay correction factor
E = detector efficiency
I = ingrowth correction factor
V = sample volume or weight
T = sample count time
R = chemical recovery
A = emission abundance

b) Radiochemistry laboratories shall also calculate a sample-specific MDA for each radiochemical parameter. MDA values shall be calculated according to the equation below and reported with each analytical result submitted to the SNL/SMO. This calculation means that if we counted a sample containing net activity a large number of times, and if the mean result of those counts comes out equal to the MDA, then the result of a subsequent count would have a 5-percent probability of coming out below the L_c. This is the net concentration “which may be *a priori* expected to lead to detection” on a single measurement according to Curie (“Analytical Chemistry,” Volume 40, Number 3, March 1968, pages 586 through 593). The contract-required MDAs are provided in Attachment 2.

$$MDA = \frac{4.65(TBC)^{1/2} + 2.71}{2.22DEIVTRA}$$

Where, the variables are defined in the same way as those in the L_c calculation of item (a).

c) Low background MDA and L_c

For low-background detectors (when at least 35 background counts cannot be obtained), laboratories shall use the equations given below to calculate MDA and L_c. These equations are based upon a blank population approach to determining signal variability, used in the case for which the standard Poisson distribution assumption in the Curie equations is inappropriate due to the low number of background counts.

Laboratories must accumulate data for each parameter, matrix, approximate count time, and digestion/separation procedure to develop blank populations. “Outlier” data should be identified and excluded from blank populations. A single blank population may be used for any digestion/separation process that is common to multiple matrices, provided that reagent volumes and counting times are comparable. As noted in Section 3.6.2 of this SOW, laboratories are not to use sand or any other matrix substitute in radiochemistry preparation blanks (PBs) associated with Sandia work. As a consequence, the digestion reagents and separation processes involved in the method define the blank type.

$$\mathrm{MDA} = \frac{4.65S_g + 2.71}{2.22DEIVTRA}$$

$$L_c = \frac{2.33S_g}{2.22DEIVTRA}$$

In these equations, the variables in the denominators are defined in the same way as those in the L_c calculation of Section 3.3.4(a). S_g is the standard deviation of the blank counts, for which the equation is given below.

$$S_g = \sqrt{\frac{\sum_{i=1}^{n}(C_i - AC)^2}{n-1}}$$

Where:

C_i = blank counts – background counts
AC = average of the C_i
n = the number of blanks in the population

For the low background blank population MDA and L_c approach, laboratories shall count batch blanks on randomly chosen detectors. Each blank shall be subtracted for the current background of the detector it is counted on, with the resulting data (C_i) saved to a file that is specific to the parameter, matrix, approximate count time, and digestion/separation process. At approximately the beginning of each month, the data in those files will be used to calculate new S_g values, which in turn will be used to calculate the MDA and L_c values for that month. The C_i in the data files will be updated monthly to include only data for the 20 most recent blanks of each type.

When using the low background blank population MDA approach, laboratories will discuss that fact in the case narrative of the associated data reports.

d) Radiochemical analytical results shall be reported as measured. That is, the laboratory shall report all results, regardless of concentration or sign, and shall not report any result as “less than the MDA or L_c.” Results shall not be reported as ND. Reported results less than the MDA or total uncertainty (see below) shall be qualified with a “U” flag.

3.3.5 Analytical uncertainty

Radiochemical analytical results shall be accompanied by sample-specific uncertainty bounds that reflect the 95 percent confidence level. The uncertainty bounds shall include not only the measurement counting error, but also a technique-specific error term that includes uncertainty values for each contributing measurement process, and a sample-specific contribution reflecting specific chemical recoveries, detectors used, etc. Laboratories shall examine error contributions such as detector calibration, tracer standardization error, weighing and pipetting errors to calculate their contributions to uncertainty. All radiochemical result uncertainties shall incorporate terms for technique-related and sample-specific measurement errors.

The general form of the 1σ counting uncertainty (CU) equation is:

$$\mathrm{CU}(\mathrm{cpm}) = \sqrt{\frac{\mathrm{CR_{smp}}}{\mathrm{T_{smp}}} + \frac{\mathrm{CR_{bkg}}}{\mathrm{T_{bkg}}}}$$

The general form of the sample 1σ Total Propagated Uncertainty (TPU) equation is:

$$\sigma_{\mathrm{Conc}}\left(\frac{\mathrm{pCi}}{\mathrm{g,L}}\right) = \left|\mathrm{Conc}\right| \times \sqrt{\left(\frac{\mathrm{CU}}{\mathrm{CR_{smp}} - \mathrm{CR_{bkg}}}\right)^2 + \left(\frac{\sigma\mathrm{Eff}}{\mathrm{Eff}}\right)^2 + \left\langle\left(\frac{\sigma\mathrm{Decay}}{\mathrm{Decay}}\right)^2 - \mathrm{or} - \left(\frac{\sigma\mathrm{Ingrowth}}{\mathrm{Ingrowth}}\right)^2\right\rangle + \left(\frac{\sigma\mathrm{branch}}{\mathrm{Branch}}\right)^2 + \left(\frac{\sigma\mathrm{Yield}}{\mathrm{Yield}}\right)^2 + \left(\frac{\sigma\mathrm{aliquot}}{\mathrm{Aliquot}}\right)^2}$$

where:

$$Decay = \exp\left[\frac{-\ln 2 \times \Delta T}{HL}\right]$$

$$\sigma_{Decay} = \frac{Decay \times \ln 2 \times \sigma_{HL} \times \Delta T}{HL^2}$$

$$Ingrowth = 1 - \exp\left[\frac{-\ln 2 \times \Delta T}{HL}\right]$$

$$\sigma_{Ingrowth} = \frac{\left(1 - Ingrowth\right) \times \ln 2 \times \sigma_{HL} \times \Delta T}{HL^{2}}$$

Note: The aliquot uncertainty includes weighing, pipetting, and dilution uncertainty.

The following abbreviations are used in the above calculations. All uncertainties are at the 1σ (68%) confidence level.

Aliquot	Size of sample aliquot (in grams, liters, etc.)
$\sigma_{aliquot}$	Uncertainty of the sample aliquot
Branch	Branching ratio
σ_{branch}	Uncertainty in the branching ratio
CR_{smp}	Gross count rate (counts per minute [cpm]) of the sample target analyte (sample + background count rates)
CR_{bkg}	Background count rate (cpm) of the sample target analyte
Decay	Factor used to correct for target analyte radioactive decay
σ_{decay}	Uncertainty of the decay factor
Eff	Counting efficiency of the detector
σ_{Eff}	Uncertainty of the detector counting efficiency
HL	Half-life (min) of the target analyte
σ_{HL}	Uncertainty of the half-life (min)
Ingrowth	Factor used to correct for target analyte radioactive ingrowth
$\sigma_{ingrowth}$	Uncertainty of the ingrowth factor
T_{bkg}	Count duration (min) of the background count
T_{smp}	Count duration (min) of the sample count
ΔT	Time difference (min) between two events used in decay and ingrowth equations
Yield	Chemical yield of the target analyte
σ_{yield}	Uncertainty of the chemical yield

3.3.6 Quality Control exemption for filters

Various filter materials will be submitted for analysis. The matrix spike (MS) and replicate sample analysis requirements in this SOW 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. However, the reanalysis requirements for certain QC failures will be waived where insufficient sample remains. A detailed discussion of that condition shall be included in the case narrative when it is encountered.

3.3.7 Quality Control exemption for physical parameters

Acidity, alkalinity, BOD, color, corrosivity, dissolved oxygen, gravimetric oil and grease, hardness, ignitability, pH, titrimetric sulfide, specific conductance, all of the solids methods, and turbidity are exempt from the general inorganic QC requirements. These analyses shall be controlled according to the method QC and/or the laboratory's QC policies.

3.3.8 Batch Quality Control requirements

The replicate and spike requirements given in this SOW apply to samples submitted by Sandia. Laboratories shall not substitute replicate and spike data that were acquired for samples submitted by other clients. If multiple Sample Delivery Groups (SDGs) from Sandia are batched together, then the relevant QC data must be included in each SDG report for validation purposes. QC data acquired outside of the SDG shall not be included on the EDD.

3.3.9 Additional requirements for fluoride run by Ion Chromatography

Laboratories that run fluoride by IC must add eluent to all standards and samples to smooth the baseline at the "water ditch" and/or use a column that separates the ditch from the fluoride peak.

3.3.10 Method detection limit elevation due to internal standard contamination

When a stable isotope analog of a target compound is used as an internal standard, the possibility exists for contamination of the extract with the associated target compound. This contamination may result in a concentration for the target analyte that is sufficient to produce an unacceptable probability of false positive detections. These situations can be identified when peaks for the target compound are observed in continuing calibration blanks. When this situation occurs, the temporary solution is to elevate the MDL for the affected analyte by an amount equal to the highest observed concentration in the continuing calibration blanks containing the contaminated internal standard. If instrument sensitivity is inadequate to determine the contamination concentration, a "best guess" should be used to elevate the MDL until an uncontaminated standard can be acquired. An investigation must be initiated to determine and correct the source of the contamination so that the MDL can be restored to the original value. Prior to elevating the MDL and reporting the data, the laboratory must attempt to contact the client to discuss the situation and gain approval, however, data package delivery should not exceed the due date for this issue. In the event that the client cannot be contacted before it is necessary to report the data package, the elevated MDL must be thoroughly discussed in the case narrative.

3.4 General inorganic analytical Quality Control requirements

Situations that make meeting the QC requirements given in this SOW difficult or impossible will arise from time to time. One such example might be replicate or spike failures where a filtered water sample contains a precipitate that cannot be brought back into solution by warming to ambient temperature and agitation. A laboratory representative should contact the SDR to request an exemption from the reanalysis requirement for QC failures that are believed to result from unavoidable inhomogeneity or other issues relating to the nature of the sample matrix or available volume.

3.4.1 Calibration verification

Required calibration verification data are the ICVs and CCVs.

a) ICV for general inorganic analysis is conducted immediately after the instrument has been calibrated. This verification consists of analysis of a standard solution within the range of calibration. The ICV standard shall be from a source different from that used to prepare the calibration standards.

b) CCV for general inorganic analysis is conducted every 2 hours or after every tenth analytical sample, whichever is more frequent. The same standard used for calibration may be used for the CCVs. The CCV standard and a calibration blank shall be analyzed at the end of each analysis run. [The term “analytical sample” refers to all samples run other than calibration standards, calibration verifications, and calibration blanks. All method blanks or PBs, spiked samples, laboratory control samples (LCSs), Reporting Limit Verification for ICP-AES and ICP-MS Methods (CRI)/Reporting Verification for AA Methods (CRA)/Method Detection Limit Verification (MDLV) samples, interference check samples (ICS) (Solution A and Solution AB), and laboratory replicates are analytical samples. However, replicate burns in GFAA work are considered to be one analytical sample.]

c) ICV and CCV results shall be within ± 10 percent of the known value. Exceptions are cyanide (± 15 percent), perchlorate by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (± 15 percent), and mercury (± 20 percent).

d) In the event that either the ICV or CCV data fall outside of these limits, the instrument shall be recalibrated and all of the samples run since the last successful calibration verification shall be reanalyzed for the failed parameters.

e) No instrument calibration is employed in the methods exempted in Section 3.3.7 of this SOW. These analyses are exempt from the instrument calibration verification requirements. However, the iodine solution used in sulfide analysis shall be calibrated against a certified titrant of known normality at least once a week. The results of iodine solution calibration shall be recorded on the chemist's worksheet, but need not be reported in the QC summary.

3.4.2 Calibration blanks

Initial calibration blanks (ICBs) and CCBs shall be run immediately following the associated calibration verification samples. The calibration blank matrix is the same as that of the calibration verification sample; that is, if the calibration standards and verification samples are digested, then the calibration blanks are also digested.

a) Calibration blanks are run with the same frequency as calibration verifications.

b) If the absolute value of the blank result for general inorganic parameters exceeds two times the MDL, the analysis shall be terminated and the problem corrected. Recalibration followed by calibration verification and blank samples shall be performed prior to resuming the analytical run.

3.4.3 Preparation blanks

PBs consisting of DI water and the appropriate reagents are included in each batch of samples requiring digestion or distillation. One PB shall be included for every 20 samples or one per batch, at a minimum.

a) PB analysis is applicable to all analyses requiring sample preparation prior to analysis, except those cases for which reagents are automatically added to all samples by an autoanalyzer. In the latter case, the ICB is equivalent to a PB.

b) If the absolute value of any analyte concentration in the blank exceeds 1/2 the PQL, the lowest reported concentration in the associated samples must be at least 10 times the concentration in the blank. All samples having that analyte's concentration at less than 10 times that of the associated value of the blank but above the PQL shall be redigested and reanalyzed.

3.4.4 Interference check samples

a) ICSs for ICP-AES and ICP-MS analyses shall be run at the beginning of each analysis run. The constituent composition of the ICSs is specified in SW-846 methods 6010 and 6020. The Sandia analytes not covered by the SW-846 methods shall be spiked into the ICS-AB solutions at 1 mg/L for ICP-AES and 0.02 mg/L for ICP-MS. The true values for ICS analytes may be calculated if diluted from certified materials.

b) The results for the trace (non-spiked) analytes in the ICS (Solution A) should be evaluated. If the absolute value of any analyte exceeds two times the MDL, the analyte should be noted in the case narrative.

c) The results for the analytes in the ICS (solution AB) shall agree within ± 20 percent of the true value. If this criterion is not met, the analyst may either terminate the analysis or continue and run the failed constituents at a later time. Analyte data obtained during an analytical run for which the ICS result does not pass the above criterion shall not be reported to the SNL/SMO.

3.4.5 Serial dilution

One serial dilution analysis shall be performed for each matrix in every batch for ICP-AES and ICP-MS analyses. The analysis is accomplished by diluting the sample(s) by a factor of five and comparing the dilution-corrected results to those for the undiluted sample(s). The serial dilution results shall agree to within ± 10 percent of the undiluted sample results where the undiluted results are greater than or equal to 10 times the PQL. Results that fail the acceptance criterion shall be qualified with an “E” when reported. No acceptance criterion applies when the undiluted sample results are less than 10 times the PQL. While this departs from the Method 6020 requirement of 100 times the reagent blank concentration, it should be much easier to implement in practice.

3.4.6 Linear range verification

In accordance with the 2004 NELAC standard, quarterly linear range verification samples may not be used to justify reporting ICP-AES and ICP-MS analytical results that exceed the calibration range. All samples that exceed the concentration of the high standard will be diluted to within the calibration range. Alternatively, laboratories may run a linear range verification sample in batches containing over-calibration samples. To be acceptable, linear range verification sample results must agree with the known values within ± 10 percent of the known value. Obviously, it is in the laboratories’ best interest to design calibrations such that they cover as much of an instrument’s linear dynamic range as possible. All analytical results, other than ICP-AES and ICP-MS results as described here, must be acquired within the calibration range.

3.4.7 Laboratory control samples

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

a) Two exceptions to the LCS requirements are mercury in water and cyanide in both soil and water. Since the ICV is always digested for these analyses, it is equivalent to an LCS. However, solid reference materials are available and should be used as LCS samples for Hg in soil analyses.

b) Analytical results for aqueous LCS shall agree within ± 20 percent of the true value for all general inorganic parameters. The control limits shall be included in the QC portion of the deliverable.

c) Solid LCS materials shall be run with each batch of solid samples when such reference materials are available. Solid LCS results shall fall within the control limits specified by the agency that prepared the reference material or statistically derived limits. If a reference material certificate includes acceptance ranges, statistically derived limits shall not exceed those specified on the certificate. Under no circumstances shall a solid LCS be used when the applicable acceptance criteria exceed the 30- to 150-percent recovery range. The laboratory shall include the control limits for solid LCS standards in the QC portion of the deliverable.

d) If the LCS data fail to meet the specified acceptance criterion, the analysis shall be terminated and the samples associated with that LCS shall be redigested and reanalyzed.

3.4.8 Replicate analyses

One replicate sample shall be analyzed from each batch, with a minimum frequency of one per 20 samples. If the SW-846 method explicitly requires the analysis of a matrix spike duplicate (MSD), such as is true in method 6010B, then the MSD analysis can replace the replicate requirement. In that case, the precision criteria given in this section still apply. Some SNL projects may elect to allow or require MSD analyses to replace replicate analyses. The SDR should be contacted for direction on this point.

a) The replicate relative percent difference (RPD) is the measure of precision used for all general inorganic constituents. The RPD is calculated as follows:

$$RPD = \frac{|S - R|}{(S+R)/2} \times 100\ \%$$

Where:

RPD = relative percent difference
S = sample value (original sample or MS value)
R = replicate value (or MSD value)

The RPD shall be less than or equal to 20 percent for samples with concentrations greater than or equal to 5 times the PQL. For samples with concentrations less than 5 times the PQL but greater than the PQL, the control limit is ± PQL. No precision criterion applies to samples with concentrations less than the PQL. If MS/MSD is used as the replicate analysis, then the RPD shall not be calculated using percent recovery but must be calculated using the measured concentrations. The RPD control limits shall be included in the QC portion of the deliverable.

b) If the above criteria are not met for filtered water samples, or solid samples that have been crushed and homogenized, all samples in the analytical batch must be redigested and reanalyzed. If the replicate precision criteria are not met in the second analysis, the results associated with the best replicate result shall be reported and qualified with the “*” flag as specified in Section 4.1.10(e) of this SOW. For unfiltered water samples and solid samples that have not been crushed and homogenized, results associated with a failed replicate analysis may be qualified and reported without reanalysis.

c) Samples identified as field or equipment blanks shall not be used to satisfy the replicate analysis requirement.

3.4.9 Spiked sample analyses

MS analyses are performed as a measure of the ability to recover analyte. As with replicate analyses, the minimum frequency is one per batch or one per 20 samples, whichever is more frequent. Matrix spike duplicates shall be performed when requested on the ARCOC. Matrix spike duplicates shall be reported with a calculated RPD as described in Section 3.4.8. If required by particular methods, post-digestion spikes will also be analyzed.

The percent recovery for spiked samples is calculated as follows:

$$\% \text{ Recovery} = \frac{SSR-SR}{SA} \times 100\ \%$$

Where:

SSR = spiked sample result
SR = sample result
SA = spike added

a) Matrix spikes shall be performed for all analytes except sodium, potassium, magnesium, calcium, and the parameters listed in Section 3.3.7 of this SOW. In addition to the exceptions listed here, aluminum and iron spikes are not required for soil samples.

b) If the control criteria given below in item (f) are not met for MS results for filtered water samples, or solid samples that have been crushed and homogenized, all samples in the analytical batch must be redigested and reanalyzed. If the control criteria are not met for the second MS analysis, the results associated with the best MS analysis shall be qualified “N” and reported.

A post digestion spike (PDS) shall be run for unfiltered water samples and solid samples that have not been crushed and homogenized with results associated with a failed MS analysis. Post digestion criteria are the same as that for the MS. Both MS and PDS shall be reported.

c) For IC, ion specific electrode, and colorimetric techniques for which no digestion is employed, analytical spikes shall be analyzed. If an analytical spike result is outside the control criteria specified below in item (f), all samples associated with the analytical batch shall be reanalyzed. If the control criterion is not met for the second analytical spike, the results associated with the best of the two spike analyses shall be qualified “N” and reported.

d) The spiking levels shall be at approximately the mid-point of the calibration range except as noted in item (e) below.

e) Laboratories running As, Cd, Pb, Sb, Se, and Tl by axial-viewing ICP-AES or ICP-MS shall spike at the concentrations given below.

As	40 ppb
Cd	5 ppb
Pb	20 ppb (water)/100 ppb (soil)
Sb	100 ppb
Se	10 ppb
Tl	50 ppb

f) The MS recovery control limits are 75 to 125 percent. An exception to these control limits is made in the case for which the sample result exceeds 4 times the spike added. No control limits are applied in this case, since the spike signal rapidly becomes negligible with respect to the sample analyte signal. However, the analysis of post digestion spikes may be required under these circumstances for some projects.

g) Samples identified as field or equipment blanks shall not be used to satisfy the spike analysis requirement.

3.4.10 Reporting Limit Verification analyses

Reporting Limit Verification (RLV) standards are run at the beginning of each inorganic analysis run as a measure of accuracy near the reporting limit. For sequencing purposes, the RLVs shall be considered analytical samples. RLV standards are prepared with concentrations at approximately the PQL. If the low calibration standard is run at a level appropriate to the RLV, then the calibration standard signal may be reprocessed against the new calibration curve instead of running a separate standard. The results for these analyses are reported on Contract Laboratory Program (CLP) Form II (Part 2) or equivalent. The acceptance criterion for these analyses is 80- to 120-percent recovery. If the RLV recovery is outside the control limit, the chemist must terminate the analysis, initiate corrective action, and successfully reanalyze the RLV before proceeding.

To demonstrate sensitivity and accuracy for perchlorate by LC/MS/MS at the MDL, a MDLV standard at two times the MDL concentration is run at the beginning and end of each analysis run. For sequencing purposes, the MDLV shall be considered an analytical sample. The MDLV must recover within ± 30 percent. If an initial MDLV recovers outside the control limits, the chemist must terminate the analysis, initiate corrective action, and successfully reanalyze the MDLV before proceeding. If a final MDLV fails, the chemist must correct the problem and reanalyze all samples from that batch whose results were at or below twice the MDL concentration.

> **Note**: It is important to remember that in LC/MS/MS perchlorate analysis, the isotope ratio value is tightly controlled as a fundamental part of the method. That means that the minor ion signal must be large enough to allow for reliable analyte identification (via the isotope ratio), and hence the

meaning of “MDL” in this context differs from the definition in 40 CFR 136.

3.4.11 Internal standards for general inorganic analyses

a) It is strongly recommended that internal standards (usually yttrium or scandium) be used in all ICP-AES work to compensate for possible transport effects.

b) Internal standards are required for ICP-MS. The method guidance provided in SW-846 Method 6020A (Rev. 1, February 2007) Section 9.6 shall be followed, except the control criteria for internal standard intensities shall be 60 - 125 percent. If the intensity of any internal standard falls outside these limits, the dilution procedure described in SW-846 Section 9.6 for internal standard matrix effect shall be followed.

3.4.12 Perchlorate analysis

Perchlorate is normally requested by EPA Method 314 with results confirmed by LC/MS/MS, SW-846 Method 6850 modified. When results are equal to or greater than the MDL of 4 ppb, the samples shall be reanalyzed using the LC/MS/MS method. Prior to the reanalysis, the laboratory shall contact the SDR for specific directions.

Daily calibration shall be performed when perchlorate is run by LC/MS/MS. Calibration shall consist of a blank and five standards, with curve definition by linear regression. The minimum acceptable correlation coefficient for calibration curves is 0.995. To be acceptable, the concentration corresponding to the absolute value of the calibration curve’s Y-intercept must not exceed 50 percent of the detection limit value.

The use of an ^{18}O labeled perchlorate internal standard is recommended to assess method performance and better quantify the analyte. If the measured ^{18}O perchlorate internal standard area (or concentration) in any sample varies from the initial calibration value by more than 50 percent, the sample must be run at increasing dilutions until the ± 50 percent acceptance criterion is met.

Perchlorate analyses performed by LC/MS/MS should include the use of two barium cartridges and one hydrogen cartridge for cleanup. However, this requirement can be waived if the laboratory can demonstrate sufficient chromatographic separation of the analyte from interfering species. As an example, some researchers report that perchlorate retention times greater than 13 minutes, combined with diversion of the anion matrix to waste during the first 10 minutes, can reduce or eliminate the need for sample pretreatment. Whether sample cleanup is used or not, the laboratory should recover a standard from a matrix containing 500 ppm each of chloride, sulfate, carbonate, and bicarbonate in every batch. The concentration of this standard should be at the PQL, which is defined to be 5 times the detection limit concentration. To demonstrate that perchlorate is adequately isolated and recovered under the specific conditions used, this standard should recover within ± 20 percent of the known value.

The transitions associated with both the ^{37}Cl and ^{35}Cl perchlorate ions (m/z 101 to 85 and m/z 99 to 83 transitions) should be monitored in perchlorate analyses. Tandem mass spectrometry is required for this analysis.

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. Isotope ratio acceptance criteria should be derived using a population of perchlorate spikes in real sample matrices, QC and test sample matrices, and some DI water matrices (not DI water alone). A variety of perchlorate concentrations throughout the calibration range should be included in the isotope ratio data population. The mean of the ratio population should not deviate by more than 10 percent from the 3.08 theoretical value, and the standard deviation should not significantly exceed 0.2. Between the detection limit and the PQL, the individual sample isotope ratio control limits should be near the population mean ± 20 percent (approximately 3σ). Above the PQL, the individual sample isotope ratio control limits should be near the population mean ± 15 percent (approximately 2σ).

Analytical results that fail the isotope ratio acceptance criteria should be flagged with a qualifier to denote the existence of “presumptive evidence suggesting that the reported analyte is not present in the sample.” When using such a qualifier, laboratories must provide supporting data and explanatory case narrative comments in the data package.

Recommended soil preparation is as follows: Weigh 2 grams of sample into a clean centrifuge tube. Add 20 milliliters of ASTM Type I water and agitate on a wrist shaker for 30 minutes. Centrifuge for 30 minutes, and then draw off 10 mL for cleanup (Ba and H cartridges) and analysis.

3.4.13 Uranium isotope analysis

Uranium isotopes are normally requested by alpha spectrometry. While this method allows the measurement of all uranium isotopes, the uranium-238/uranium-235 ratios tend to be biased due to uranium-234 overlap into the uranium-235 region of interest. This is not the case with ICP-MS analysis. When uranium isotopes are requested by ICP-MS, the laboratory will use the radiochemistry total digestion, including HF, unless specifically exempted by the requesting program. Uranium isotope reporting will include the reporting of the uranium-238/uranium-235 ratio. Results shall be reported as activity concentration and not as mass concentration.

The equations for uranium mass to activity conversion are:

$$\text{pCi } ^{235}\text{U} = (\text{ug } ^{235}\text{U})/22.0$$

$$\text{pCi } ^{238}\text{U} = (\text{ug } ^{238}\text{U})/3.33$$

3.5 Organic analytical and Quality Control requirements

Organic analytical and QC requirements are specified in this section of the SOW. The laboratory shall follow the requirements specified in SW-846 or other EPA methods as requested by the SNL/SMO. Additional general analytical and QC criteria are specified in Sections 3.5.1 through 3.5.10 of this SOW. Method-specific analytical requirements are given in Section 3.5.11. The SNL/SMO's ongoing laboratory performance assessments will be based partially upon the quality of the chromatography achieved.

3.5.1 Required target analytes and target MDLs

The target analytes and target MDLs for each method are specified in Attachment 3.

3.5.2 Instrument calibration

Unless otherwise specified in the method or this SOW, GC, GC/MS, LC/MS/MS and high performance liquid chromatography (HPLC) instrument calibration shall be performed using a minimum of five calibration standards, with the low standard at or below the PQL. All GC/MS instruments shall be tuned according to the frequency and ion abundance requirements of the applicable method. All initial calibrations will be verified using a second-source calibration verification standard. Laboratories may (1) verify the calibration using a second-source standard immediately after the initial calibration, (2) use a second-source CCV standard with each analytical run, or (3) use a second source LCS. If a second source LCS is used, the LCS must contain all of the compounds in the initial calibration, and the acceptance criteria shall be those specified for the CCV. Full list LCS samples shall be analyzed and reported unless instructed differently. Exceptions are designated below in Section 3.5.11.

a) <u>Calibration acceptance criteria</u>

Method-specific calibration criteria are specified in Section 3.5.11 of this SOW and in the analytical methods. In the absence of method-specific calibration acceptance criteria, the general calibration acceptance criteria are:

i. The percent relative standard deviation (percent RSD) for the response factors (RFs) obtained from the five or more initial calibration standards should be less than 20 percent unless otherwise specified in the method. In accordance with the new method 8000C, RSD averaging shall not be used. If the initial calibration fails these criteria, the laboratory shall follow the calibration guidance given in method 8000C.

ii. The percent difference of the daily or continuing calibration standard RF (or Calibration Factor [CF]) from the average RF (or CF) obtained from the initial calibration must be within ± 20 percent unless otherwise specified in the method. CCV recovery averaging shall not be used. If calibration verifications fail these criteria, the laboratory shall take corrective action and perform a new initial calibration. As specified in SW-846 Method 8000C, GC and HPLC work will include a CCV sample at both the beginning and the end of each analytical run. GC/MS work will include a calibration verification analysis at least once every 12 hours. The laboratory may request exemption from this requirement for individual compounds that are known "poor performers." In this case, a compound-specific criterion should be proposed that would be used instead of the 20-percent criterion. Exemptions should not be requested on a batch basis.

Laboratories need not include all the compounds from the initial calibration if some compounds are not requested as target analytes. However, compounds that are not considered in the CCV evaluation must be listed in the case narrative as having been excluded.

iii. As described in SW-846 Method 8000C, laboratories may use least-squares regression to generate linear calibration curves, provided the correlation coefficients for the resulting curves are at least 0.990. Forcing the resulting curves through zero is not recommended. However, if the curves are forced through zero, correlation coefficients that are ≥ 0.995 must be obtained for the curves to be acceptable. Correlation coefficients may not be rounded up to achieve compliance with this requirement. If linear curves are generated, laboratories must have formal criteria addressing the concentration axis intercept. The concentration axis intercept shall be within ±3 times the MDL for all analytes subjected to linear calibration.

b) Low-concentration soil VOC analysis calibration

For SW-846 Methods 8021B and 8260C, a separate initial calibration shall be performed for low-concentration soil samples if the purge vessels or purge conditions used are different from those used for water. Medium-concentration soil extracts may be analyzed using the same purge vessels and initial calibration as those used for water samples.

3.5.3 Quantitation of optional compounds

The laboratory shall quantitate additional compounds, whether unlisted or listed as optional in Attachment 3, at the request of the SDR for a fixed price per method per compound. The laboratory shall calibrate the instrument using a single standard containing the specified non-target analyte(s). Under such circumstances, the SDR shall provide the laboratory with non-target compound standard material required for instrument calibration, or shall reimburse the laboratory for the purchase of a standard material at cost.

3.5.4 Sample preparation

a) Volatile Organic Compound analysis

For SW-846 Methods 8021B and 8260C, water and soil samples shall be prepared and purged into the GC instrument using Method 5030C or 5035A, as appropriate to the sample vessel provided and SDR contract requirements. The laboratory may employ the VOC screening procedures described in SW-846 Methods 3810 or 3820, to determine whether sample dilution is required.

b) Volatile Organic Compound soil and solid waste extractions

Any low-concentration soil sample analysis for which a saturated detector response is observed in SW-846 Methods 8021B and 8260C shall require a medium-concentration soil analysis. The smallest amount of soil sample on which a low-concentration analysis shall be performed is 1 gram. Medium- and high-concentration soil and solid waste samples shall be extracted using methanol as described in Method 5035A.

c) Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure Volatile Organic Compound extractions

For VOC TCLP or SPLP extract analyses, the laboratory shall use properly maintained and inspected zero-headspace extraction vessels, as described in SW-846 Method 1311, "Toxicity Characteristic Leaching Procedure," and SW-846 Method 1312, "Synthetic Precipitation Leaching Procedure," to extract samples.

d) Sample extraction and cleanup

Depending upon the characteristics of the sample matrix and the method requested, the laboratory shall use an appropriate method to extract samples and shall specifically reference the method used in the extraction log.

Unless otherwise specified in this SOW, the guidance in the methods shall be followed for cleanup procedures. Initial dilution of extracts to eliminate interferences is generally not allowed due to the attendant harm to surrogate recoveries and detection limits. Extracts shall be subjected to appropriate cleanup steps when visual inspection or surrogate failures indicate that significant matrix interferences exist. Appropriate cleanup methods for each determinative analytical method are listed below.

i.	SW-846 Method 8011	Method 3620C Florisil Cleanup Method 3640A Gel Permeation Cleanup (GPC) Method 3660B Sulfur Cleanup
ii.	SW-846 Method 8082	Method 3660B Sulfur Cleanup

		Method 3665 Sulfuric Acid/Potassium Permanganate Cleanup
iii.	SW-846 Method 8151	Method 8151 Sec. 7.2.4 Method 3620C Florisil Cleanup
Iv.	SW-846 Method 8270	Method 3640A Gel Permeation Cleanup (GPC) Method 3611B Alumina Column Cleanup

3.5.5 Sample analysis acceptance criteria

The acceptance criteria for organic analyses are specified below. Samples for which the analyses fail to meet these criteria shall be reanalyzed by the laboratory at no additional cost to the SNL/SMO.

a) Sample extraction and analysis, confirmation of detection, and any required reanalysis must be performed within the holding times specified in Attachment 5.

b) The retention time of the surrogate compounds and any detected target analytes must be within the retention time acceptance windows for all columns. Unless otherwise specified in Section 3.5.11 or the analytical method, retention time windows shall be calculated using the procedure described in SW-846 Method 8000C. The retention times for all analytes in the initial CCV must be within the retention time windows established with the initial calibration.

Most of the analytical methods will include examples of the expected run times and analyte retention times for the instrumentation used in developing the method. In some cases, newer technology has allowed significant decreases in sample run times. If the laboratory implements any technology that significantly decreases the analysis time for methods that rely on retention times for all or part of the analyte identification, the laboratory must include demonstration of adequate resolution and identification of compounds that elute at similar retention times when one compound has a low concentration (at about the MDL) and one is high (at about the concentration of the high standard). In addition, retention time criteria must be adjusted accordingly to account for the shorter analytical runs.

c) Surrogate recovery acceptance criteria should be calculated according to the guidance given in SW-846 Method 8000C. The resulting calculated acceptance criteria should be within or near those given in Attachment 6 of this SOW. Laboratories will derive skewed acceptance limits if the results for nonroutine matrices (e.g., sludge for soil populations) are included in the populations from which these statistics are derived.

Similarly, routine failure to employ the method-specified extract cleanup procedures will skew the acceptance limits. The laboratory must consciously avoid these circumstances. Reported recoveries shall be accompanied by the applicable acceptance limits. If surrogate recoveries fail the acceptance criteria, the sample(s) shall be reanalyzed, typically after performing extract cleanup steps. If the surrogates fail in the second analysis, both results shall be reported and discussed in the case narrative. If the surrogates for the second analysis pass, the successful analysis results shall be reported. All analyses with target analytes reported shall have surrogates reported from that analysis.

d) A saturated detector response for target compounds must initiate dilution and reanalysis for those compounds.

e) The concentration of target analytes in the solution being analyzed must not exceed the concentration of the high calibration standard. When sample dilutions are required, chromatographic peaks chosen to quantify target analytes must be reported at between 10 and 100 percent of full scale. The scaling factor used must appear on all chromatograms. Appropriately scaled chromatograms must be provided in data reports for all dilutions for which data are reported.

f) The additional method-specific sample analysis acceptance criteria given in Section 3.5.11 must be met.

g) Manual integration must not be performed solely for the purpose of meeting QC criteria. Eliminating part of the subject peak area or including peaks not belonging to the subject peak are inappropriate manipulation of the analytical data. The laboratory must provide full explanation and documentation, including the original and manual integration, for every occurrence of manual integration, and for every analyte affected. Manually integrated data must be clearly indicated and must always include documentation, including “before” and “after” areas, clearly stating the reason the manual integration was performed, who completed the work, and the initials of the peer reviewer, group leader, or QA coordinator reviewing the manual integration.

3.5.6 Blank analysis

a) <u>Method blank (preparation blank) analysis</u>

The laboratory shall run a Method Blank (MB) for all methods at a frequency of once per delivery order, once per 20 analytical samples, once per sample matrix, or at the frequency specified in the method, whichever is more frequent. MBs for VOC analyses shall consist of reagent water that has been taken through the same preparation steps (as applicable) as those used for samples. For SW-846 Methods 8081B, 8082A, 8151A, and 8270D, soil MBs shall consist of a reagent blank only; the use of artificial matrices such as anhydrous sodium sulfate is strongly discouraged. MB acceptance criteria are given below in item (d). Samples associated with an unacceptable MB analysis shall be reanalyzed at no additional cost to the SNL/SMO. Method blank analyses must be performed in exactly the same manner as the samples: on the same instrument as all samples, and within 72 hours of all samples.

b) <u>Instrument blank analysis</u>

An instrument blank, including internal standards when the method calls for internal standard calibration, shall be run after each CCV, ICV or analysis of a sample or sample dilution that contained a target compound in greater concentration than the initial calibration range or other contaminant that saturated the instrument's detector. Blanks shall be run in the same purge inlet position (if applicable) as was the contaminated sample, and must meet the blank acceptance criteria given in item (d) below. If a blank fails the blank acceptance criteria, the instrument shall be decontaminated and additional blanks run in the same purge inlet port (if applicable) until the blank acceptance criteria are passed.

It is not practical to insert blanks for automated analytical runs. When a compound has been observed above the calibration range or a large peak was observed in the chromatogram, the sample immediately following should be carefully evaluated for carryover. If carryover is observed, the sample must be reanalyzed at no additional cost to the SNL/SMO.

c) <u>Storage blank analysis</u>

For SW-846 methods 8021B and 8260C, laboratories shall prepare storage blank samples and store these in the appropriate sample storage area. Each storage blank sample shall consist of a 40-mL screw-cap volatile sample vial having a Teflon®-lined septum and filled with reagent water. One storage blank shall be run at the end of each subsequent week. Target analytes measured above the associated PQL shall be reported by telephone and/or fax to the SDR within 24 hours, and discussed in the case narrative of reports for samples stored during the applicable period. The storage blank reporting requirement is waived in the case for which all Sandia samples stored during the period were analyzed and showed no target analyte “hits.”

d) Blank acceptance criteria

The acceptance criteria for all blank analyses are given below.

i. All sample analysis acceptance criteria for the specific analytical method were met.

ii. The concentration of each target analyte found in the blank must be less than the associated MDL. This blank acceptance criterion is waived in two cases: when Sandia samples show no target analyte “hits” on compounds detected in the blank, or when the associated sample results are ≥ 10 times the blank contaminant’s concentration. In either of these circumstances, the rationale for accepting the contaminated blank must be discussed in the case narrative.

If the concentration of any compound in a blank exceeds the PQL, and the results for that compound in the associated samples also exceed the PQL, then the lowest reported concentration in the associated samples must be at least 10 times the concentration in the blank. All samples having that compound’s concentration at less than 10 times that of the blank but above the PQL shall be re-extracted and reanalyzed.

e) Solvent blanks

It is expected that chemists will run solvent blanks when they encounter conditions that could adversely affect analytical work by causing carryover, causing baseline rise, etc. However, laboratories serving the SDR should not make a routine practice of running solvent blanks immediately prior to or following CCVs or other QC analyses. This practice suggests that analytical conditions are not adequate to eliminate memory effects for analytes that are within the calibration range and is not allowed. In isolated cases, solvent blanks may need to be run immediately before QC samples. The reasons for running a solvent blank before a QC sample must be discussed in the associated instrument run logs. In no case, shall multiple CCBs or solvent blanks be run immediately after a CCV.

3.5.7 Matrix Spike and Matrix Spike Duplicate analyses

a) The laboratory shall perform MS and MSD analyses for all methods except TO-13A and TO-14A at a frequency of once per delivery order, once per 20 samples, or once per sample matrix, whichever is more frequent. The laboratory shall use a Sandia sample and shall not use field blank, equipment blank, or trip blank samples to satisfy this requirement.

b) MS and MSD analyses must meet all sample analysis acceptance criteria. Unless otherwise specified in Section 3.5.11, 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. Calculated QC acceptance criteria shall not exceed those found in specific SW-846 methods. Laboratories will report recoveries and RPD values for MS and MSD analyses in the QC section of deliverables.

c) Laboratories shall use a full list spiking solution except as specified in Section 3.5.11. The laboratory may request exemption from this requirement for individual compounds that are known "poor performers." This is intended for implementation in the case for which a chemical incompatibility exists between calibration compounds, such as is true for benzidine. When spiking compounds are excluded, those compounds shall be listed in the case narrative.

3.5.8 Laboratory Control Sample analysis

An LCS shall be analyzed by the laboratory for all methods at a frequency of once per delivery order, once per matrix, or once per 20 analytical samples, whichever is more frequent. LCS analyses must be performed in exactly the same manner as the samples: on the same instrument as all samples, and within 72 hours of all samples. LCS analyses must meet all sample acceptance criteria. QC acceptance criteria for LCS results shall be derived statistically by each laboratory for each method using the procedure given for QC check samples in SW-846 Method 8000C unless specific criteria are given in the SW-846 method. All samples associated with an unacceptable LCS analysis, as described in the next paragraph, shall be re-extracted and reanalyzed at no additional cost to the SNL/SMO.

If used for second-source calibration verification, the LCS shall be prepared from standard materials that are independent of those used for calibration and contain all of the analytes in the initial calibration (see Section 3.5.2 of this SOW). Laboratories shall use a full list spiking solution except as specified in Section 3.5.11. The laboratory may request exemption from this requirement for individual compounds that are known "poor performers." This is intended for implementation in the case for which a chemical incompatibility exists between calibration compounds, such as is true for benzidine. When spiking compounds are excluded, those compounds shall be listed in the case narrative. Laboratories must use the following criteria when establishing LCS acceptance criteria.

a) The LCS must contain (at a minimum) the same analytes as the MS samples.

b) The concentrations of the LCS compounds shall be near the mid-point of the calibration range. The laboratory shall calculate data acceptance criteria using the procedure for QC check samples given in SW-846 Method 8000C.

c) The LCS acceptance criteria must not exceed those specified in the method.

d) Without special exemption, the recovery control limits shall not be less than 10 percent and not be greater than 150 percent.

e) If the LCS fails, corrective action (re-extraction and reanalysis) must be performed regardless of the outcome of the MS and MSD analyses. See exceptions in item (f) below.

f) LCS analytes may marginally fail the LCS acceptance criteria without initiating corrective action; however, all LCS analyte failures MUST be documented and discussed in the applicable case narrative(s). A marginal failure is defined as greater than three standard deviations but less than four standard deviations from the mean. If a large target analyte list LCS is analyzed, the following criteria may be used for LCSs that fall outside reported acceptance criteria but have >10 percent recovery:

70 to 74 compounds	5 LCS compounds may fall outside acceptance criteria with no corrective action.
60 to 69 compounds	4 LCS compounds may fall outside acceptance criteria with no corrective action.
50 to 59 compounds	3 LCS compounds may fall outside acceptance criteria with no corrective action.
40 to 49 compounds	2 LCS compounds may fall outside acceptance criteria with no corrective action.
30 to 39 compounds	1 LCS compound may fall outside acceptance criteria with no corrective action.
< 30 compounds	No LCS compounds may fall outside acceptance criteria with no corrective action.

The guidelines above should not be applied to matrix spikes. If the guidelines given above for target compound acceptability are not met, laboratories may contact the SDR to request special exemption allowing them to report the data. Such requests will be evaluated individually by the SNL/SMO.

3.5.9 Second-column or Gas Chromatography/Mass Spectrometry confirmation

Second-column or GC/MS confirmation of compound identification is required where recommended by the method. Laboratories may use a single-standard calibration passing through the origin for Method 8330 CN confirmation columns. For GC methods requiring second-column confirmation (routinely 8081, 8082, and 8151), the calibration requirements of this SOW and the applicable SW-846 methods shall be met on both columns. All confirmation results must be reported as part of the QC summary and must include estimated (8330A HPLC) or quantified (GC methods) concentrations for confirmed compounds. In addition, confirmation analyses must be discussed in the case narratives of the applicable deliverables.

Laboratories must conduct MDL studies on the columns or separate instruments used in confirmation analyses. The MDL reported for an analysis requiring second-column or GC/MS confirmation must be the higher of those obtained on the primary column and confirmation column (or instrument).

Compounds that are detected on the primary column but not detected on the confirmation column must be discussed in the case narrative. If the RPD between primary and confirmation column results is greater than 40 percent, that fact must also be discussed in the case narrative and the associated data must be qualified with a “P” flag.

3.5.10 Process artifacts

Process artifacts (such as aldol condensates) and column degradation products (siloxanes) identified in Sandia samples shall be discussed in the case narrative in addition to any data qualification requirements.

3.5.11 Method-specific analytical requirements

The additional analytical requirements given below are organized by SW-846 method. The target analyte lists to be used are provided in the attachments of this SOW.

a) Petroleum hydrocarbons by Gas Chromatography/Flame Ionization detector (FID)

Unless otherwise specified by the SNL/SMO, petroleum hydrocarbon analysis shall be performed using a modified SW-846 Method 8015D. Regardless of the method specified, laboratories shall adhere to the QC requirements given in this SOW, SW-846 Method 8000C, and SW-846 Method 8015D. At the request of the SNL/SMO, the instrument may be calibrated for petroleum hydrocarbons based on a range of molecular weights or product type (such as GRO), or calibrated using a specific petroleum product (such as Fuel Oil No. 2). The capability to identify specific petroleum products that may be present in samples is desired but is not a requirement.

i. Analysis of a CCV, a retention time marker standard and blank is required at least once in each 12-hour period. The blank must be analyzed after the standard analyses.

ii. Modified 8015D method analyses are exempt from the sample acceptance criteria requiring extract cleanup and reanalysis based upon surrogate recovery (see Section 3.5.4.(d) in this document).

iii. It is not always possible to insert blanks for automated analytical runs. When a compound has been observed above the calibration range or a large peak was observed in the chromatogram, the sample immediately following should be carefully evaluated for carryover. If carryover is observed, the sample must be reanalyzed at no additional cost to the SNL/SMO.

b) Organochlorine pesticides by Gas Chromatography

Unless otherwise specified by the SNL/SMO, organochlorine pesticide analysis shall be performed according to the requirements listed in SW-846 Method 8081B, "Organochlorine Pesticides by Gas Chromatography." Regardless of the method specified, laboratories shall adhere to the QC requirements given in this SOW, SW-846 Method 8000C, and SW-846 Method 8081B. Method 608 may be requested. When method 608 is requested, the laboratory will perform 8081B but report the analyte list for Method 608.

i. All soil sample extracts shall be subjected to the Florisil cartridge cleanup procedure described in SW-846 Method 3620C. Water samples shall also be subjected to the Florisil cleanup prior to reporting when MS or surrogate results fail the acceptance criteria.

ii. Soil, sediment, and biological sample extracts shall be subjected to the GPC cleanup procedure described in Method 3640A when MS or surrogate results fail the acceptance criteria. In addition, all water samples containing high molecular weight compounds that interfere with analysis of the target compounds must undergo GPC cleanup.

iii. All sample extracts that are contaminated with elemental sulfur shall be subjected to the sulfur cleanup procedure described in Method 3660B.

iv. If the system is primed prior to analysis, a solvent blank should be run before any standards or samples.

v. A CCV for multi-component analytes must be analyzed during a valid analytical sequence on the same instrument, column, and calibration within 72 hours of its detection in a sample. If the CCV fails, the extract shall be reanalyzed against a new calibration. If the instrument was not previously calibrated for the detected multi-component analyte, the extract shall be reanalyzed against a new calibration.

vi. Confirmation of all target analytes detected above the MDL is required. All calibration and method QC criteria must be met on the confirmation column used. If the RPD between the results obtained on the primary and confirmation columns is greater than 40 percent, the lower of the two shall be reported on the Form I.

vii. An LCS containing the organochlorine pesticides shall be analyzed for every 20 samples or every batch, whichever is more frequent. If chlordane and/or toxaphene are target analytes, an LCS shall be prepared and run for the appropriate multi-component analyte. LCS data acceptance criteria shall be derived by the laboratory according to the procedure for QC check samples given in SW-846 Method 8000C.

viii. Degradation problems shall be checked by injecting a standard containing only 4,4'-DDT and endrin. Presence of 4,4'-DDE, 4,4'-DDD, endrin aldehyde, or endrin ketone indicates breakdown. If degradation of either DDT or endrin exceeds 15 percent, corrective action must be taken before proceeding. Unless otherwise specified by the SNL/SMO, this test should be performed even when DDT and endrin are not target analytes for a given project.

c) Polychlorinated Biphenyls by Gas Chromatography

Unless otherwise specified by the SNL/SMO, Polychlorinated Biphenyl (PCB) analysis shall be performed according to the requirements listed in SW-846 Method 8082A. Regardless of the method specified, laboratories shall adhere to the QC requirements given in this SOW, SW-846 Method 8000C, and SW-846 Method 8082A.

i. Sulfuric acid (H2SO4)/potassium permanganate (KMnO4) cleanup (SW-846 Method 3665A) is strongly recommended for all sample extracts. Modification of the cleanup procedure to neglect the KMnO4 step is acceptable, provided that H2SO4 alone produces acceptable results. However, laboratory SOPs should provide instruction on the KMnO4 step, anticipating that the additional oxidizer will sometimes be needed.

ii. Sulfur cleanup, Method 3660B, shall be used when extracts are contaminated with elemental sulfur. Sequential cleanup steps shall be used if necessary to eliminate the interference.

iii. Laboratories shall routinely report the seven target Aroclors for this analysis. However, if the target PCB congeners are requested, decachlorobiphenyl shall be used as an internal standard by adding it to each calibration standard and sample extract, including QC samples. In this latter case, tetrachloro-meta-xylene is used as a surrogate.

iv. When these compounds are determined as aroclors, decachlorobiphenyl shall be added to each sample extract as a surrogate.

v. SW-846 Method 8082A is exempt from the full list LCS requirements discussed in this SOW. Laboratories may use a short list of aroclors, such as 1016 and 1260, for this purpose.

vi. Confirmation of all target analytes detected above the MDL is required. All calibration and method QC criteria must be met for the confirmation column used. If the RPD between the results obtained on the primary and confirmation columns is greater than 40 percent, the lower of the two shall be reported on the Form I.

vii. If an initial calibration has not been performed for any target Aroclor for which a detected result above the MDL is obtained, the laboratory must calibrate for that Aroclor and reanalyze the extract. If an existing initial calibration has not been verified via CCV for any target Aroclor for which a detected result above the MDL is obtained, an acceptable CCV run must be obtained within 72 hours, at a minimum, if the result is to be reported.

d) Chlorinated herbicides by Gas Chromatography

Unless otherwise specified by the SNL/SMO, chlorinated herbicide analysis shall be performed according to the requirements listed in SW-846 Method 8151A. Regardless of the method specified, laboratories shall adhere to the QC requirements given in this SOW, SW-846 Method 8000C, and SW-846 Method 8151A.

i. The laboratory shall use 2,4-dichlorophenylacetic acid (DCAA) as a surrogate standard to monitor the performance of the method's extraction and analysis steps. DCAA shall be added to standards, blanks, and all analytical samples. If DCAA is expected to be present in samples, the laboratory shall use a chlorinated herbicide not present in the samples as the surrogate compound. In this case, the laboratory should consult the SDR on the selection of the surrogate compound.

ii. Sample cleanup shall be performed, as needed, to eliminate sample interferences using SW-846 Method 8151A Section 7.2.4, or the Florisil cartridge cleanup procedure described in Method 3620C.

iii. Confirmation of all target analytes detected above the MDL is required. All calibration and method QC criteria must be met for the confirmation column used. If the RPD between the results obtained on the primary and confirmation columns is greater than 40 percent, the lower of the two shall be reported on the Form I.

e) Volatile organic compound analysis by Gas Chromatography/ Mass Spectrometry

Unless otherwise specified by the SNL/SMO, VOC analysis shall be performed according to the requirements listed in SW-846 Method

8260C, “Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry.” Regardless of the method specified, laboratories shall consider the QC requirements given in the SOW, SW-846 Method 8000C, and SW-846 Method 8260C to be the minimum requirements. If EPA Method 524.2, Method 624, or a 25-mL purge is requested, the laboratory must determine whether the Method 8260 analyte list is adequate to cover the site target analyte list. If a 25-mL purge is used, the laboratory must discuss that fact in the case narrative.

i. The concentration of methylene chloride in blank analyses must be less than 2.5 times the required PQL, and acetone and 2-butanone must be less than 5 times their required PQL. If these concentration limits are exceeded, laboratories shall discuss the blank contamination in the associated case narrative.

ii. No quantitation ion may saturate the instrument’s detector. When this occurs, decontamination procedures must be employed as necessary to demonstrate that the system is free from contamination. If a blank has not been analyzed because the analytical run was automated, the subsequent sample must be reanalyzed if the same compound(s) that was over the calibration range in the previous sample was detected.

iii. The laboratory may be asked to tentatively identify and report up to 20 of the non-target compounds having the greatest apparent concentration in the sample and whose response is greater than 10 percent of the nearest internal standard. These compounds shall be tentatively identified and quantified following the guidelines provided within the specific analytical method being used.

iv. Laboratories shall use an industry standard spectral library to obtain reference spectra. Instrument-generated quality factors (Q factors) indicating spectral ion abundance match with library reference data shall be presented on the quantitation reports for all target compounds and tentatively identified compounds (TICs).

v. Per Section 11.4.7 of SW-846 Method 8260C, if the extracted ion current profile (EICP) area for any of the internal standards in the calibration verification standard changes by a factor of two (-50 percent to +100 percent) from that in the mid-point standard level of the most recent initial calibration sequence, the purge and trap sampler and GC/MS system must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, samples analyzed while the system was malfunctioning must be reanalyzed.

f) Semivolatile organic compound analysis by Gas Chromatography/ Mass Spectrometry

Unless otherwise requested by the SNL/SMO, semivolatile organic compound (SVOC) analysis shall be performed according to the requirements listed in the SW-846 Method 8270D, “Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry.” Regardless of the method specified, laboratories shall consider the QC requirements given in the SOW, SW-846 Method 8000C, and SW-846 Method 8270D to be the minimum requirements.

i. For soil, sediment, and biological samples, GPC cleanup using SW-846 Method 3640A or Alumina cleanup using SW-846 Method 3611B shall be used as necessary to eliminate interferences. In addition, all water samples containing high molecular weight compounds that interfere with analysis of the target compounds must also undergo GPC cleanup.

ii. As stated in SW-846 Method 8270D, the minimum average RF for all target analytes shall be 0.05. These criteria also apply to daily CCV standards.

iii. Laboratories may request permission to use statistical process control criteria for a small number of poorly performing CCV compounds. If granted, these criteria must be present on recovery reports for CCV samples or otherwise clearly presented in data deliverables. No individual recovery value shall exceed a ± 60 percent control criterion, and each individual target compound that recovers outside the ±20 percent window must be called out in the case narrative.

iv. Target phthalate esters are exempt from the reanalysis requirements associated with MB contamination up to a concentration of five times the PQL.

v. No quantitation ion may saturate the instrument’s detector. When this occurs, decontamination procedures must be employed as necessary to demonstrate that the system is free from contamination. If a blank has not been analyzed because the analytical run was automated, the subsequent sample must be reanalyzed if the same compound(s) that was over the calibration range in the previous sample was detected.

vi. The laboratory may be asked to tentatively identify and report up to 30 of the non-target compounds having the greatest apparent concentration in the sample and whose response is greater than 10 percent of the nearest internal standard. These compounds shall be tentatively identified and quantified following the guidelines provided in the specific analytical method being used.

vii. Laboratories shall use an industry standard spectral library to obtain reference spectra. Instrument-generated Q factors indicating spectral ion abundance match with library reference data shall be presented on the quantitation reports for all target and Tentatively Identified Compounds (TICs).

viii. Per Section 11.4.7 of SW-846 Method 8270D, if the extracted ion current profile (EICP) area for any of the internal standards in the calibration verification standard changes by a factor of two (-50 percent to +100 percent) from that in the mid-point standard level of the most recent initial calibration sequence, the GC/MS system must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, samples analyzed while the system was malfunctioning must be reanalyzed.

g) Polychlorinated dioxins and furans by Gas Chromatography/Mass Spectrometry

Unless otherwise specified by the SNL/SMO, all sample analysis shall be performed according to the requirements listed in SW-846 Method 8290A.

i. No quantitation ion may saturate the instrument’s detector. When this occurs, decontamination procedures must be employed as necessary to demonstrate that the system is free from contamination. If a blank has not been analyzed because the analytical run was automated, the subsequent sample must be reanalyzed if the same compound(s) that was over the calibration range in the previous sample was detected.

h) Nitroaromatics and nitramines by High Performance Liquid Chromatography

Nitroaromatics and nitramines analysis shall be performed according to the requirements specified in SW-846 Method 8330B. Laboratories shall adhere to the QC requirements of this SOW, SW-846 Method 8000C, and SW-846 Method 8330B.

i. The laboratory shall use an appropriate surrogate compound that does not co-elute with any target analytes on the C18 column to monitor the performance of the analytical method. To minimize co-elution problems on both columns, it is recommended that either 1,2-dinitrobenzene or 1,4-dinitrobenzene be used as the surrogate.

ii. All target analytes detected in samples shall be confirmed on a secondary column. Secondary columns should provide a separation that is substantially different from that obtained on the primary column. The estimated analyte value, obtained using at least one standard to calibrate the secondary column, shall be reported for confirmation analyses. Co-elutions are prohibited in secondary column calibrations. Laboratories shall only report the results obtained from the C18 column on the Form I, regardless of the RPD between the C18 and secondary column results.

iii. Solid phase extraction (SPE) is approved for HE extractions in water samples. Laboratories must perform an initial demonstration of proficiency using SPE that includes a maximum concentration per target compound and a maximum total concentration for HE target compounds. These maximum values must be set by procedure and must trigger re-extraction on a smaller sample aliquot if exceeded. Laboratory procedures must also address sample filtration to prevent reduced extraction efficiency resulting from particulates clogging the extraction media.

SPE approval applies to groundwater and surface water; application to matrices with high organic content may be inappropriate. Laboratories should demonstrate the efficacy of SPE individually, per matrix, for effluent, or waste sample matrices. Laboratories may select an early-eluting surrogate for addition to the method to indicate breakthrough or elution caused by organic substances in the samples. Laboratories must separate the phases of multi-phasic samples prior to extraction.

iv. Laboratories shall analyze and report CCVs and MBs in secondary column confirmation runs. The analysis frequency requirements for analyses using the secondary column are the same as those used for the primary column.

v. Limited spectral information is available if a diode array detector is used. This detector approach is preferred by the SDR because of the increased ability to identify false positives that it affords. However, this does not replace the requirement for secondary column confirmation.

vi. The comments of this section apply to analyte detections that have been confirmed on a secondary column. If LC/MS/MS is to be used as an additional confirmation for HPLC 8330B, the instrument will be calibrated according to the guidance in SW-846 Method 8321B and Method 8000C

1. Laboratories shall use at least two internal standards for this HE technique. In any sample analysis, the area of the internal standard peaks must be within ± 30 percent of the average internal standard areas from the initial calibration. Also in any sample analysis, the retention time of the internal standards must be within ± 30 seconds of the average internal standard retention times from the initial calibration. If internal standard areas vary by

> ± 30 percent , and/or internal standard retention times vary by more than ± 30 seconds from the average values of the most recent initial calibration sequence, the LC/MS/MS system must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, samples analyzed while the system was malfunctioning must be reanalyzed.

2. MDLs reported for LC/MS/MS will be at such a level as to have signal-to-noise ratios of approximately five or higher.

3. CCVs and CCBs shall be analyzed at the beginning of each analytical run, at least once every 10 analytical samples, and at the end of each analytical run. If calibration verification follows a new initial calibration, then an ICV replaces the first CCV and must be a second-source standard. The ICV and CCV acceptance range is ± 20 percent. When LC/MS/MS is the primary measurement technique for high explosives, CCV acceptance criteria are as follows:

   - When CCV recovery for any compound exceeds +/- 20 percent, the associated field sample data for that compound should be qualified, with the failure discussed in the case narrative. Reanalysis is not required.

   - If the CCV recovery for any compound exceeds +/- 30 percent in any two consecutive CCVs, significant drift is indicated. Laboratories must take the appropriate corrective action (typically recalibration), but need not reanalyze the field samples associated with the ICV/CCVs. The affected field sample data shall be qualified and the failure shall be called out in the case narrative.

   - If the CCV recovery for any compound exceeds +/- 50 percent, the laboratory shall take the appropriate corrective action and shall reanalyze all field samples back to the most recent CCV for which the recovery was within +/- 30 percent.

4. The laboratory may use the same extract as that used for the HPLC analysis. In that case, except as directed in the section below, the laboratory need not prepare any additional batch QC if the PB results, surrogate recoveries, and LCS results from the HPLC analysis are acceptable.

5. For all confirmation runs, the laboratory will add an analytical spike to at least one sample that was determined in HPLC analysis to have detections at or near

the associated MDL. The analytical spike concentration will likewise be at or near the MDL. Acceptable recoveries for the analytical spike are between 70 and 130 percent of the known value.

6. The case narrative will discuss the LC/MS/MS confirmation results, and the associated LC/MS/MS data will be placed in the miscellaneous data section of the deliverable.

7. Target analytes that are detected by HPLC (both columns) but do not confirm by LC/MS/MS will still be reported. However, such results will be flagged with an "X" qualifier. All uses of the "X" qualifier must be explained in the case narrative.

8. By definition, analytes must be detected on both the C18 and the secondary columns to be reportable detections in SW-846 Method 8330B analyses. In LC/MS/MS confirmation analyses, results for analytes that have not met this criterion shall not be reported.

9. The extract holding times for SW-846 Method 8330B that are given in Attachment 5 of this SOW apply.

10. Only LC/MS/MS methods are acceptable for this work because of the high specificity of the ion transitions that are monitored for each compound. These transitions are unique to a particular molecular structure and are crucial to compound identification in this analysis. LC/MS/MS methods that do not monitor such transitions shall not be used.

vii. If HE compounds are to be analyzed by LC/MS/MS as the primary technique, then the internal standard, calibration, MDL, and calibration verification requirements of LC/MS/MS confirmation analysis apply. In this work, laboratories shall use the same extraction procedures that are used for SW-846 Method 8330B HPLC analyses.

1. To demonstrate sensitivity and accuracy for the analysis of HE by LC/MS/MS at the MDL, an MDL verification (MDLV) standard at approximately two times the MDL concentration is run with every CCV during the analytical run. The MDLV must recover within 30 percent for every compound. If any compound recoveries from the MDLV analysis are outside the control limits, the chemist must correct the problem and reanalyze all samples from that batch.

2. Surrogate recovery, MS, and LCS acceptance criteria shall

be separately established via statistical process control for HE by LC/MS/MS. The LCS and MSs shall contain the full list of target analytes.

3. MS samples shall be prepared at concentrations that result in an extract concentration that is near the midpoint of the calibration range for each target analyte and shall be analyzed at least once per batch.

4. All holding times for SW-846 Method 8330B that are given in Attachment 5 of this SOW apply.

5. As in the confirmation protocols discussed above, only LC/MS/MS (tandem quadruple) methods are acceptable for this work.

i) Volatile organic compounds in ambient air using Gas Chromatography/Mass Spectrometry

Analysis shall be performed according to the requirements specified in EPA Method TO-14A, revision 1.0.

i. A GC/MS analytical system shall be used.

ii. Canisters obtained from the laboratory shall be certified as containing less than 0.2 parts per billion by volume (ppbv) VOCs through humid zero air analysis.

iii. An acceptable daily humid zero air instrument blank shall be analyzed immediately prior to and after instrument calibration. These instrument blanks must be less than 0.2 ppbv for all target analytes before analysis may proceed.

iv. MS and MSD analyses are not required for this method.

v. The LCS shall contain all of the target analytes at concentrations near the mid-point of the calibration range. Recovery for the target analytes must be within ± 20 percent of the theoretical value.

vi. Laboratories shall use a minimum of three surrogate compounds and report the resulting surrogate recovery data with EPA Method TO-14 QC deliverables.

3.6 Radiochemistry analytical Quality Control requirements

Standards used in batch QC analyses, such as LCS and spiking standards, need not be NIST-traceable. Standards requiring NIST-traceability are discussed in Section 3.6.9 below.

3.6.1 Calibration verification

Calibration verification samples and calibration blanks are not required for radiochemistry. This statement refers only to batch QC and in no way diminishes the calibration requirements given in Section 3.6.9 of this SOW.

3.6.2 Preparation blanks

One PB shall be included for every 20 samples or one per batch, at a minimum. An empty or water-filled container for the appropriate geometry shall be run for gamma spectroscopy. <u>Laboratories shall not use silica sand or any other matrix substitute in PBs for solid sample analyses</u>. Artificial urine may be used in PBs for urine sample analyses.

a) PB analysis is applicable to all analyses requiring sample preparation prior to analysis. An aliquot-specific preparation blank report shall be provided, in which PB results are calculated assuming aliquot sizes comparable to the sample aliquots used in the associated batch.

b) Samples associated with any PB result that is greater than its associated MDA shall be redigested and reanalyzed. Exceptions to this requirement are samples for which the measured concentrations in the samples are greater than or equal to five times the PB value. Reanalysis is not required for such samples. PB results that are greater than their associated MDAs shall not be included in any blank population calculations.

c) PBs for alpha spectrometry, GFPC, and Lucas cell techniques shall be placed randomly or sequentially, such that the blank position varies from batch to batch. Instrument run logs shall be maintained to demonstrate compliance with this requirement.

3.6.3 Laboratory control samples

LCSs shall be analyzed using the same sample preparation and analysis methods used for the Sandia samples. One LCS shall be analyzed with each batch of up to 20 samples. LCS standards shall derive from a source different from that used to calibrate the instrument.

a) <u>Solid LCS materials shall be analyzed with each batch of solid samples when such materials are available</u>. A laboratory representative may call the SDR for assistance if solid LCS materials appropriate to requested analyses cannot be obtained. Aqueous LCS standards shall be analyzed if neither the laboratory nor the SDR can obtain appropriate solid LCS materials.

b) The aqueous LCS analytical results shall agree within ± 20 percent of the true value.

c) Solid LCS results shall fall within the control limits specified by the agency that prepared the reference material or statistically derived limits developed by the laboratory. The laboratory shall include the control limits in the QC portion of the deliverable.

d) If the LCS data fail to meet the applicable acceptance criterion, all samples associated with that LCS shall be redigested and reanalyzed.

e) LCS results reported with the QC data for gamma spectroscopy shall include Am-241 (59.5 kilo electron volt [keV]), Cs-137 (661.7 keV), and Co-60 (1332 keV) at a minimum.

f) The LCS for gross alpha and gross beta by GFPC shall contain solids such that the measured mass is about the mid-point of the mass attenuation curves.

3.6.4 Replicate analyses

One replicate sample shall be analyzed from each batch, with a minimum frequency of one per 20 samples.

a) The replicate error ratio (RER) is used to determine replicate precision for radiochemical results. The RER is given by:

$$RER = \frac{|S - R|}{\sigma_{95S} + \sigma_{95R}}$$

where, RER = replicate error ratio
S = sample value (original)
R = replicate sample value
σ_{95S} = sample uncertainty (95 percent)
σ_{95R} = replicate uncertainty (95 percent)

Radiochemical replicate determinations shall agree when the 95 percent confidence level uncertainties are considered. That is, the RER shall be less than or equal to 1. This control criterion is not applied, and reanalyses or data qualification are not required, when both of the measured values are less than their associated MDAs.

b) If the RER control criterion is not met for filtered water samples, or for solid samples that have been crushed and homogenized, all samples in the analytical batch must be redigested and reanalyzed (see the exception below). If the control criterion is not met for the second replicate analysis, the results associated with the best replicate analysis shall be qualified "*" and reported. For unfiltered water samples and for solid samples that have not been crushed and homogenized, results associated with a failed replicate analysis may be qualified and reported without reanalysis.

c) Samples identified as field or equipment blanks shall not be used to satisfy the replicate analysis requirement.

d) Replicate analyses may not be possible in soil tritium analysis when the moisture content is too low or the sample size is too small. A discussion of this problem shall be included in the case narrative if tritium replicates cannot be run.

e) Circumstances occasionally preclude adequate homogenization of samples. Examples of this are some plutonium analyses and samples from areas where depleted uranium munitions have been used. Laboratories that believe the reanalysis requirement should be waived in a specific case due to unavoidable inhomogeneity should seek SDR approval for suspension of the reanalysis requirement.

3.6.5 Spiked sample analyses

MS analyses are performed on field samples as a measure of the ability to recover analytes. As with replicate analyses, the minimum frequency is one per batch or one per 20 samples, whichever is more frequent.

a) If a MS result is outside the control criterion specified in item (d), all samples associated with the analytical batch shall be redigested and reanalyzed. If the control criterion is not met for the second MS analysis, the results associated with the best MS analysis shall be qualified “N” and reported. As in the section above addressing replicate analyses, 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 criterion may be qualified and reported without reanalysis.

b) Matrix spikes are not required for gamma spectroscopy, Rn-222, or any analyses utilizing a tracer that is chemically identical to the analyte. Matrix spikes are likewise not required for analyses that utilize a standard addition for every sample (such as is commonly done for ^{3}H and ^{99}Tc). In addition, Ra-226 analyses that employ a Ba-133 tracer are exempt from the MS requirements.

c) Sample spiking levels for radiochemical analyses other than tritium shall be added at a concentration of at least 5 but not greater than 20 times the estimated MDA. Tritium samples must be spiked before the distillation step, at a level chosen by the laboratory.

d) The spike recovery control limits are ± 25 percent. An exception to these control limits is made in the case for which the sample result exceeds 4 times the spike added. No control limits are applied in this case.

e) Samples identified as field or equipment blanks shall not be used to satisfy the spike analysis requirement.

f) The considerations of Sections 3.6.4 (d) and (e) may also apply to the MS analysis and reanalysis requirements. The actions recommended in those sections should be followed if applicable.

3.6.6 Chemical recovery requirements for radionuclides

a) Correction of analytical results for radionuclide chemical recovery shall be performed sample specifically unless the SDR has given prior approval for a batch-correction procedure. Carrier recovery shall be corrected for the indigenous carrier concentration of the element in the sample.

b) Recovery guidelines for tracer and carrier results in routine matrices (soil and water) shall be 50 to 105 percent. The SDR is aware that the tracer recovery requirements cannot be met for some difficult matrices. Recoveries that do not meet the acceptance criteria given in this paragraph must be approved by the SDR prior to submission of the deliverable. If reanalysis is requested and the resulting tracer recoveries still do not pass the criterion given here, the laboratory shall report the batch having the best recoveries and discuss the results and corrective actions in the case narrative.

The tracer recovery criteria in this section exist to ensure that detection limits are not deleteriously affected by low recoveries and that analytical results that are corrected for those recoveries are not excessively uncertain (see below). In general, the SDR should approve minor variances from these criteria. More significant variances will be evaluated in the context of counting uncertainty for the tracer and the detection limits that are achieved. If an unacceptable uncertainty has not been introduced, and if the required detection limit has been meet, then the SDR should approve reporting the results without reanalysis.

c) The concentration of tracer material added shall be sufficient to result in a maximum of 5 percent uncertainty in the measured chemical recovery at the 95 percent confidence level, and at the recovery level expected for the matrix and method. This means that at least 400 counts should be acquired for tracers.

3.6.7 Blank subtraction

Blank subtraction shall be done only in liquid scintillation counting. Results for the other counting techniques shall be corrected for instrument background only, and shall not be blank subtracted.

For liquid scintillation, there are three blanks. The detector background is measured with the blank from the vendor's QC set, sample results are subtracted for calibration blank results, and random contamination is identified and reported via the PB results.

- The data from the vendor's blank are used to assess instrument background.

- The calibration blank contains the cocktail and any reagents added to the batch, and is placed in a vial from the same lot used for the samples, but is not subjected to the separation or distillation steps/apparatus. The calibration blank is used to determine the background for a particular batch of samples. This result is subtracted from all the samples in the batch.

- The PB is used to identify contamination from sample preparation processes. PBs are made in the same way the calibration blanks are, but are additionally subjected to the same separation or distillation steps used for the samples. This result is reported as PB and is not subtracted from each sample result.

3.6.8 Target Critical Levels (concentration corrected) and Minimum Detected Activities

The tables in Attachment 2 give target critical levels corrected for concentration of radionuclides by analytical technique and matrix. Laboratories shall adjust analytical conditions to meet the target L_c.

For gamma spectroscopy, Cs-137, Co-60, and Am-241 shall be reported for every sample. Analytical conditions shall be adjusted to meet the specified MDAs for those radionuclides. The analytical conditions chosen will determine the MDAs for other reported nuclides.

3.6.9 Counting instrument calibration requirements

Counting instruments are subject to a primary calibration prior to initial use, when the instrument is placed back in service after repair, and when the instrument's performance parameters exceed previously established acceptance criteria. Radionuclide analyses that do not involve nuclear disintegrations are defined to be general inorganic analyses. Such analyses are subject to the analytical and QC requirements in the appropriate section of this SOW. This applies to total and isotopic uranium determination by ICP-MS.

Primary calibration shall be performed using NIST-traceable standards except where such standards are unavailable. The words "check" and "verification" below apply to measurements performed to verify the primary calibrations. Standards used for this purpose shall be independent of the primary calibrants, and shall also be NIST-traceable or have been directly compared with NIST standards. If such verifications fail, the laboratory shall reassess all data acquired since the last successful check and notify the SDR if corrections are made.

a) <u>Gas flow proportional counting</u>

   i. Background counts equal in duration to the longest expected sample count time and used for background corrections shall be performed at least quarterly.

   ii. Daily background checks shall be performed.

iii. Calibration for detector efficiency shall be performed when daily checks (see below) fail the laboratory's acceptance criteria.

iv. Detector efficiency checks shall be performed daily before use.

v. Mass attenuation curves shall be generated at instrument setup, following major maintenance, and when detector efficiency check failures necessitate recalibration. Enough standards must be used to adequately define the curves, and at least 10,000 counts must be acquired for each calibration standard. Mass attenuation curves shall be constructed for each detector unless the laboratory can show that all detectors, or designated groups of detectors, are statistically equivalent.

vi. Voltage plateau performance checks shall be made after each gas bottle change or maintenance activity.

vii. Cross talk determinations shall be verified at least annually.

viii. Laboratory calibration procedures shall require that backgrounds be checked after counting high-activity samples.

ix. Gross Alpha/Beta Planchette Flaming: Gas proportional counting in the presence of hygroscopic salts, which thicken as they absorb moisture, inhibits alpha transmission. Flaming to red heat converts the slats to oxides which are less likely to absorb alpha particles. However, flaming to red heat drives off volatile elements (which tend to be beta emitters. Most notable losses to volatility will be cesium (Beta), polonium (alpha), and technetium (Beta)).

Gross alpha/beta is intended as a screening method that provides qualitative information. When gross alpha/beta results are used as a screening tool, prepared planchettes maintained in a desiccated environment should not require flaming prior to analysis. When gross alpha/beta results are used for regulatory purposes, or certain geological studies, a higher level of certainty is required. In these instances, a sequence of beta count, flame to dull red heat, alpha count should be employed. In all cases, the case narrative must identify when flaming is employed and specify the counting sequence associated with the flaming. This will clearly identify any possible limitations of the gross alpha/beta results. Laboratories shall have a formal procedure for the count/flame/count option available for use at the discretion of the SNL/SMO.

b) <u>Alpha spectrometry</u>

i. Background counts equal in duration to the longest expected sample count time shall be performed at least monthly.

ii. Energy/channel calibrations shall be verified at least weekly.

iii. Detector efficiency shall be verified at least monthly.

iv. Laboratory calibration procedures shall require that backgrounds be checked after counting high-activity samples.

v. Refer to Section 3.3.4(c) of this SOW for the blank population approach to detection limit calculation for low background detectors.

c) Gamma spectroscopy

i. Calibration background counts equal in duration to the longest expected sample count time shall be performed at least monthly, with verification performed weekly. Use of the MB for weekly background checks is acceptable, provided that the data are compared to the original calibration background.

ii. Energy/channel calibrations shall be verified on the day of use.

iii. Efficiency calibrations shall be verified on the day of use.

iv. Resolution calibrations shall be verified on the day of use.

d) Liquid scintillation

i. Daily verification checks shall be performed using a vendor supplied unquenched standard set (H-3, C-14 and blank).

ii. Each batch shall contain a calibration blank vial to be used for blank subtraction (Section 3.6.7 of this SOW).

iii. If the constant quench method of calibration is used, the quench of the sample shall be within +/- 5 percent of the quench of the efficiency standard. If this condition is not met, the sample must be reanalyzed beginning with a new sample aliquot.

e) Kinetic Phosphorescence Analysis for uranium

Kinetic Phosphorescence analysis (KPA) has been found to be unreliable due to strong susceptibility to interferences from constituents commonly found in Sandia samples. KPA shall not be used in the analysis of Sandia samples. ICP-MS is the preferred technique for total uranium determinations.

f) Alpha scintillation (Ra-226 by Rn emanation)

i. The efficiency of detector/cell combinations (cell constants) shall be determined at least annually, with verification monthly or after maintenance activities.

ii. Detector/cell background shall be measured before counting each sample.

3.6.10 Reporting non-target radionuclides in gamma spectroscopy

The laboratory shall report any and all non-target radionuclides having activities greater than the minimum detectable concentration (MDC) that are identified using the gamma spectroscopy software without any additional charge to the SNL/SMO. Care will be taken to ensure that these non-target radionuclides are not interference from NORM or other high-activity radionuclides.

3.6.11 Reporting K-40 for soils in gamma spectroscopy

Laboratories must report the result for K-40 with those for the target analytes when soil samples are analyzed. If K-40 is not identified in any Sandia soil sample, the laboratory must include a discussion of the reasons for that fact in the case narrative. In general, corrective action will be required if the laboratory fails to identify K-40 in soil samples.

3.6.12 Reporting Radium-226 in gamma spectroscopy

The laboratory shall follow the provided guidelines when reporting radium-226 results by gamma spectroscopy.

a) Quantification using radon-222 daughter analytes

If lead-214 and/or bismuth-214 results are to be used to report radium-226 in a solid sample matrix, the sample must be prepared according to Section 3.2.2 of this SOW, along with the requirement that the sample container be completely filled with sample (i.e., no head-space) and tightly sealed such that radon gas cannot escape. The detector must be calibrated with a standard that matches this geometry. The sealed sample must undergo a 21-day ingrowth period before gamma count to allow radon-222 daughters to reach secular equilibrium with radium-226. If radium-226 is reported without meeting these conditions, it must be documented in the case narrative that the radium-226 sample result is estimated with suspected negative bias due to incomplete equilibrium with daughter nuclides.

Note: Though it is possible to report radium-226 in an aqueous matrix using the above guidelines, the more sensitive analysis by Lucas Cell is typically employed to satisfy required detection limits.

If the above conditions are met, lead-212 and/or bismuth-212 can be used to report radium-224 and/or thorium-228.

b) Quantification using the radium-226 186 keV emission line

The guidelines in Section 3.6.12 (a) are preferred for reporting radium-226 by gamma spectroscopy. For samples that have reporting turnaround times that disallow 21-day ingrowth periods, the radium-226 186 kilo electron volt (keV) emission line can be used to report radium-226 results if the following conditions are met. If a uranium-235 peak is observed at 144 keV (and possibly at 163 keV and 205 keV), the 186 keV peak must have the appropriate number of uranium-235 counts subtracted from it before it is used to quantify radium-226. The radium-226 uncertainty and MDC results must be adjusted for this interference correction. If uranium-235 is not observed in the gamma spectrum, radium-226 can be reported using the 186 keV peak without correction.

3.6.13 Tritium distillations

Soil tritium determinations shall be performed using vacuum or cryogenic distillation. The aliquot shall be taken to complete dryness during the distillation process to ensure complete removal of titrated water. For very dry samples, a second aliquot may have to be distilled. Dead water should only be added to the distillates with SDR approval and documented in the narrative.

3.7 Asbestos analysis

3.7.1 Accreditation

Laboratories must be accredited by the AIHA to be eligible to perform airborne asbestos analysis for the SNL/SMO. Laboratories must participate in and report results to the SDR for all PE rounds to demonstrate that the accreditation is current. In addition, the SDR must receive copies of each report, response, and close-out letter for audits performed by the accrediting agency.

3.7.2 Staff qualifications

Individuals performing the preparation and phase-contrast microscopy analysis of airborne asbestos filters shall have successfully completed the NIOSH 582 course. Individuals analyzing bulk samples shall have successfully completed the McCrone Research Institute course in polarized-light microscopy identification and quantitation of asbestos minerals in bulk samples.

3.7.3 Quality control

a) Laboratories performing airborne asbestos analysis shall conform to the requirements of the accrediting agency, including participation in the AIHA Airborne Asbestos Proficiency Analytical Testing (PAT) program and the interlaboratory sample exchange program. In addition, archived PAT program samples shall be analyzed with every sample batch and reported with the batch results. The known values and acceptance windows provided by the PAT program shall be used as acceptance criteria. The laboratory QA officer or his/her designee shall periodically re-label the known samples so that they are submitted as blinds to the analyst.

b) Laboratories performing bulk asbestos analysis shall conform to the requirements of the AIHA, including participation in the AIHA Bulk Asbestos PAT Program. Participation in the NIST National Voluntary Laboratory Accreditation Program for bulk asbestos is also recommended. Laboratories are encouraged to retain samples from those programs and submit them as blinds with each batch as specified above for airborne samples. Required QC practices in the laboratory procedures shall include verification of microscope alignment and performance. Specific QC practices for particular asbestos types and matrices shall be determined by mutual agreement between the laboratory and the SNL/SMO.

3.8 Geotechnical analyses

3.8.1 Accreditation

Samples that the SDR typically sends to laboratories for geotechnical testing will not require American Association of State Highway Transportation Officials (AASHTO) certification. If the certification is required, the SDR will send those samples to a certified laboratory.

3.8.2 Facilities and training

Facilities and staff training levels for participating laboratories must comply with ASTM D3740, “Minimum Requirements for Agencies Engaged in Testing for Engineering Design and Construction.”

3.8.3 Methods

Laboratories shall use the methods in the most recent Annual Books of ASTM Standards to perform geotechnical tests. Laboratories shall adhere to the QC requirements given in the ASTM methods used.

3.8.4 Parameters

A list of geotechnical tests is included as Attachment 4. Analytical needs outside those listed in Attachment 4 will be addressed on a case-by-case basis.

4.0 ANALYTICAL DATA DELIVERABLE REQUIREMENTS

4.1 Analytical data package contents and format

Data packages shall contain the analytical data and appropriate supporting documentation for all samples. The standard SDR deliverable (Level C or D) shall consist of a hard copy and compact disc (CD) with an electronic file suitable for direct computer input. The electronic data files shall be provided for all sample delivery groups simultaneously with the hard copy, unless specified differently on the ARCOC. The CD shall also include an electronic copy of the complete data package. The laboratory shall provide pricing for ARCOC requests where electronic deliverables are not required. Hard copy reports shall be produced from the electronic data deliverable for consistency and compatibility. Specifications for electronic data transfer are provided by Sandia in Attachment 11, Exhibit 1. All information recorded on the hard copy report shall be legible and of sufficient print quality for normal reproduction. Each page of the report shall be sequentially numbered.

4.1.1 Level C analytical reports

Items included in Level C analytical data packages provided to the SDR are discussed in items (a) through (f) below. A comprehensive case narrative is required for all data reports submitted. Data packages shall contain all of the following items:

a) A completed Deliverable Transmittal/Review form. This form must contain the SNL ARCOC number as well as the laboratory's SDG reference.

b) A case narrative that describes the contents of the data package and provides an index of samples associated with the delivery order (including both the Sandia sample IDs and the laboratory sample IDs). A description of problems encountered in sample receipt, login, and analysis shall also be included in the narrative. The case narrative shall describe the circumstances leading to the use of data qualifiers and list the affected samples. In addition, the type of digestion used shall always be clearly specified in the case narrative for general inorganic analysis of soil samples. All case narratives shall include a signed statement affirming that the analytical work and data package have been reviewed and are in compliance with the requirements of this SOW.

c) One original Analysis Results form for each sample associated with the deliverable. The required contents of each Analysis Results form are outlined in Section 4.1.4 of this document.

d) QC data deliverables consisting of completed CLP QC data reporting forms or equivalent for all sample analyses associated with the delivery order. The QC data deliverables are discussed in detail in Section 4.1.8 of this document.

e) Signed and dated original COC forms received with each sample shipment, indicating sample receipt and custody by the laboratory. Condition-on-receipt checklist and copies of air bills shall be included.

f) One EDD of the analytical data and QC results formatted as outlined in Attachment 11, Exhibit 1, of this SOW.

g) CD with the EDD file plus an electronic copy of the data package.

Shipping documents, instrument printouts, standard preparation logs, digestion logs, analyst work sheets, or other forms of "raw" data shall not be included unless specifically requested. This material will be inspected during periodic data package assessments.

Level C reports shall be on 8.5” x 11” paper, one-sided, and paginated.

4.1.2 Level D analytical reports

a) Level D analytical report formats shall include all elements required in Level C analytical reports, as described above, plus shipping and login documents, all email correspondence applicable to the data package, analyst worksheets, instrument run logs, instrument printouts, standard preparation logs, digestion and extraction logs, and other forms of raw data as necessary to support data defensibility. Analyst worksheets and logs shall meet the minimum requirements given in this SOW. If the vendor name, lot number, and expiration date is given in tabular form on the chemist worksheets for all calibration and second-source calibration verification standards, the standards preparation logs need not be included.

b) For radiochemistry, laboratories shall adhere to the spirit of the inorganic and organic chemistry reporting requirements in preparing analytical reports. This means that laboratories performing radiochemical analyses shall include analyst worksheets, instrument printouts, standard preparation logs, digestion logs, and other forms of raw data in the reports. Raw data shall include all aliquot weights/volumes, tracer/carrier recoveries, counting times, detector efficiencies, and other information necessary to re-create analytical results. Radiochemistry counting instrument calibration data shall not be included with data reports, but rather shall be maintained by laboratories as records. However, radiochemistry data packages shall include copies of the calibration verification, blank check results, and acceptance criteria associated with the sample results being reported.

c) Standards certificate of analysis information, log entries for water quality, log entries for balance calibration verification, and other similar ancillary information shall not be included in analytical reports. Such information shall be maintained by the laboratories as records.

4.1.3 Level B analytical reports

Level B analytical reports are a simplified version of the Level C package. Level B packages will include analytical results reporting forms with a QC summary page. CLP-like forms are not required. EDD is optional. Level B reports could be requested as CD deliverables only. Laboratories shall provide pricing for Level B reports.

4.1.4 Reporting forms for analytical results

a) The Analysis Results form shall be used to report parameter concentrations measured by the laboratory. The use of CLP forms is not preferred.

b) The laboratory shall specify the complete Sandia sample ID, date of sample collection, date of sample receipt at lab, date analyzed, date extracted (where appropriate), delivery order number (SDG field), report date, analytical batch number, sample matrix, and a qualitative description of sample appearance on each page of the Analysis Results form. Alternatively, laboratories may provide sample descriptions by including sample digestion/extraction logs or a tabular summary of qualitative descriptions with the deliverable. For each result, the laboratory shall provide the parameter name, parameter value, uncertainty value (where applicable), MDL and PQL, or MDA and L_c (as applicable), units of measure, data qualifier(s), method of analysis, dilution factors (default is 1), and analysis date on the Analysis Results form. Analysis Results forms shall include the extraction date (as applicable). Alternatively, a tabular summary of extraction dates may be provided immediately following the Analysis Results forms.

4.1.5 "Less than" results

Laboratories shall not use mathematical “less than” signs in reporting Sandia analytical results. Qualifiers for low-level general inorganic and organic results are discussed in Section 3.3.3 of this SOW. Radiochemical results that are less than the MDA shall be reported as measured, with a “U” qualifier, as discussed in Section 3.3.4 of this SOW.

4.1.6 Analytical uncertainties and detection limits

The analytical uncertainty values, L_c, and MDAs for radiochemical parameters shall be reported with each result on both the hard copy and the EDD.

4.1.7 Electronic Data Deliverable format

Format requirements for the EDD copy of analytical data are provided in Attachment 11 of this SOW. The data for the hard copy deliverable and the EDD shall be drawn from the same database at the same time.

4.1.8 Reporting conventions

Anion reporting conventions are as listed below:

a) Ammonium is reported as N.

b) NO_2 + NO_3 is reported as N.

c) Nitrate is reported as N.

d) Nitrite is reported as N.

e) Total phosphorus is reported as P.

f) Sulfate is reported as SO_4.

g) Ortho-phosphate is reported as P.

4.1.9 Quality Control deliverables

a) QC data deliverables for general inorganic chemistry shall include items listed below. The delivery order number shall be given on each page of the QC data deliverable. QC acceptance limits shall be included in the QC deliverable. All QC forms shall be clearly labeled.

i. ICV and CCV analysis data shall include the parameter name, true ICV concentration, found ICV concentration, ICV percent recovery, true CCV concentration, found CCV concentration(s), and each CCV percent recovery. The use of EPA CLP Form II-IN, or an equivalent format that presents the same information, is acceptable.

ii. ICB and CCB analysis data shall include the parameter name, ICB analysis result, and CCB analysis result(s). The use of EPA CLP Form III-IN, or equivalent, is acceptable.

iii. PB analysis data shall include the parameter name and PB results for each analytical batch. The use of EPA CLP Form III-IN, or equivalent, is acceptable.

iv. ICS analysis data shall include the parameter name, true concentration values for solutions A and AB, initial measured values for solutions A and AB, initial percent recovery for solution AB, final measured values for solutions A and AB, and the final percent recovery for solution AB. The use of EPA CLP Form IV-IN, or equivalent, is acceptable.

v. Spike analysis data shall include the parameter name, spiked sample result, sample result, spike added, and spike percent recovery for each spike analysis. In addition, include the required data qualifiers for spike analyses that fall outside the control limits. The use of EPA CLP Form V (Part 1)-IN, or equivalent, is acceptable.

vi. Replicate analysis data shall include the parameter name, sample result, replicate result, and RPD. Include the required data qualifiers for replicate analyses that fall outside the applicable control limit. The use of EPA CLP Form VI-IN, or equivalent, is acceptable.

vii. LCS analysis data shall include the parameter name, true concentration of the LCS, measured concentration of the LCS, and the percent recovery for the LCS. The use of EPA CLP Form VII-IN, or equivalent, is acceptable. Solid LCS data shall be accompanied by the applicable acceptance criteria.

viii. Standard addition results shall be reported for GFAA, as appropriate. The use of CLP Form VIII-IN, or equivalent, is acceptable.

ix. Analysis run logs shall be provided. The use of EPA CLP Form XIV-IN, or equivalent, is acceptable for all parameters.

x. Initial calibration data shall be provided and shall include the number and concentration levels of calibration standards, curve equations, and correlation coefficients.

xi. RL verification (CRI and CRA) data shall be provided (Form 2B-IN or equivalent) and shall include the parameter name, true standard concentration, measured concentration, and percent recovery value.

xii. ICP-AES serial dilution data shall be provided (Form 8-IN or equivalent) and shall include for each parameter the parameter name, parameter concentration in the sample, parameter concentration in the diluted sample (corrected for the 5X dilution), and the percent difference value.

xiii. ICP-MS tune reports shall be provided and shall include for elements representing all mass ranges of interest the mass calibration values and the full width resolution values at 10 percent peak height.

b) QC data deliverables for radiochemistry shall include items listed below. The delivery order number shall be given on each page of the QC data deliverable. QC acceptance limits shall be included in the QC deliverable. All forms shall be clearly labeled.

i. The instrument calibration date and associated calibration file names shall be provided. Alternatively, this information may be placed on chemist worksheets. All calibration files shall be archived and retrievable.

ii. PB data shall be provided for each batch and shall include the parameter name, result, and uncertainty. Aliquot size corrected blank results shall be included.

iii. MS data shall include the parameter name, spiked sample result, sample result, spike added, and spike percent recovery for each spike analysis. Include the required data qualifiers for spike analyses that fall outside the control limits.

iv. Replicate data shall include the parameter name, sample result, replicate result, and RER value. Sample and replicate results for radionuclide and gross radiation determinations shall be accompanied by the 95 percent confidence level uncertainty values. Include the required data qualifiers for replicate analyses that fall outside the control limit.

v. LCS data shall include the parameter name, true concentration of the LCS, measured concentration of the LCS, and the percent recovery for the LCS. Solid LCS data shall be accompanied by the applicable acceptance criteria.

vi. The instrument and detector identifiers shall be provided for each sample. This is typically present on the instrument printouts. If so, it need not be repeated in the QC summary.

vii. Radionuclide tracer or carrier recoveries, or standard addition recoveries used for sample-specific chemical recovery correction, shall be reported in the QC deliverable. For recoveries that fail to meet the criteria specified in Section 3.6.6, a record of SDR approval to report shall be provided in the case narrative.

c) QC data deliverables for organic chemistry shall include items listed below. The delivery order number shall be given on each page of the QC data deliverable. QC acceptance limits shall be included in the QC deliverable. All forms shall be clearly labeled.

i. Initial calibration data, ICV data, and CCV data shall be presented. The initial calibration data shall include the average RF (or CF) and RSD, or the curve equations and correlation coefficients if regression is used. The calibration verification data shall include the percent difference values.

ii. Preparation or method blank data shall be provided for each batch and each 12-hour period, as applicable. The method blanks that follow CCVs in some GC methods shall be reported. Blank data shall include the parameter name and analysis result and shall be reported to the SDR on a CLP Form I.

iii. MS and MSD analysis data shall include the parameter name, spiked sample result, sample result, spike added, spike percent recovery, and RPD for each MS/MSD analysis. Include the required data qualifiers for MS/MSD analyses that fall outside the control limits. If the MS/MSD is run on a sample from another SDG, that SDG must be identified on the report.

iv. If replicate analyses are performed, the replicate data shall include the parameter name, sample result, replicate result, and RPD. Include the required data qualifiers for replicate analyses that fall outside the applicable control limits. If a replicate is run on a sample from another SDG, that SDG must be identified in the report.

v. LCS analysis data shall include the parameter name, true concentration of the LCS, measured concentration of the LCS, and the percent recovery for the LCS.

vi. Analysis run logs shall be provided for all analytical runs for which data are reported.

vii. Surrogate and internal standard recoveries, and associated acceptance criteria, shall be reported in the QC deliverable. Recoveries that fail to meet the applicable criteria shall be explained in the case narrative.

viii. Laboratories shall include Form 10 or equivalent reports to describe replicate precision and second column results for all dual-column GC and HPLC work.

ix. GC/MS tune reports shall be submitted and shall include the relative abundance values and acceptance criteria.

4.1.10 General inorganic chemistry and radiochemistry data qualifiers

General inorganic chemistry and radiochemistry data qualifiers available for use by the laboratory are listed and discussed below. The use of these data qualifiers is required on the Analysis Results form, the EDD, and the QC data deliverable. Of the qualifiers discussed below, only the “H”, “U”, “N”, “X”, and “*” may be used in reporting radionuclide and gross radiation results.

a) In the event that the holding time for a particular parameter had expired prior to analysis, flag the associated results with an "H” on the Analysis Results form and the EDD.

b) Analytical results obtained for samples that required dilution prior to analysis shall be qualified with the "I" flag. This qualifier indicates that the related detection limits are elevated due to the presence of an interference or because of a high parameter value.

c) Data associated with failed ICP-AES serial dilution results shall be flagged with the "E" flag. The "E" flag shall also be used to qualify GFAA data according to the guidelines specified in the CLP SOW. In both cases, the specific requirements of the EPA CLP SOW apply to the use of this qualifier. When this flag is used, an explanatory note shall always be included in the case narrative.

d) Analytical results associated with a spike analysis that was outside control limits shall be qualified with the "N" flag.

e) Analytical results associated with a replicate analysis that was outside the control limit shall be qualified with a "*" flag.

f) General inorganic results having concentrations between the MDL and the PQL (or RL) shall be qualified with a “J” flag.

g) The “X” qualifier is used only to denote the existence of presumptive evidence suggesting that the reported analyte is not present in the sample. That is, this qualifier may be used only to indicate that the chemist believes the result to be a false positive. When the “X” qualifier is used, laboratories must provide supporting data and explanatory case narrative comments in the data package.

h) Analytical results with associated batch QC blank concentrations greater than the MDL will be qualified with a “B” flag when the sample concentration is greater than the MDL.

i) Analytical results that are less than the MDL will be qualified with a “U” flag. For radiochemistry, results less than the MDA and/or TPU will be qualified with a “U” flag.

4.1.11 Organic chemistry data qualifiers

Organic chemistry data qualifiers available for use by the laboratory are listed below. As with general inorganic chemistry and radiochemistry, the use of these data qualifiers is required on the Analysis Results form, the EDD, and the QC data deliverable.

a) The “U” flag indicates that the compound was a target but was not detected. The result was less than the MDL.

b) The “J” flag indicates an estimated value.

    i. The “J” flag is used when estimating a concentration for TICs where a 1:1 response is assumed.

    ii. The “J” flag is used when the mass spectral and retention time data indicate the presence of a compound that meets the volatile or semi-volatile GC/MS identification criteria, and the result is less than the PQL but greater than the MDL.

    iii. The “J” flag is used when the retention time data indicate the presence of a compound that meets the GC or HPLC identification criteria, and the result is less than the PQL but greater than the MDL.

c) Analytical results associated with a spike analysis that was outside control limits shall be qualified with the "N" flag.

d) The “B” flag is used when the analyte is found in both the associated method blank and the sample. This flag indicates probable blank contamination and warns the data user to take appropriate action. This flag shall be used for both TICs and positively identified target compounds. The combination of flags “BU” or “UB” is expressly prohibited. Blank contaminants are flagged “B” only when they are detected in the sample.

e) The “E” flag identifies compounds whose concentrations exceed the upper level of the calibration range of the instrument for that specific analysis. If one or more compounds have a response greater than the upper level of the calibration range, the sample or extract shall be diluted and reanalyzed. All such compounds with a response greater than the upper level of the calibration range shall have the concentration flagged with an “E” on CLP Form I for the original analysis.

f) Analytical results obtained for samples that required dilution prior to analysis shall be qualified with the “I” flag. This qualifier indicates that the related detection limits are elevated due to the presence of an interference or because of a high parameter concentration.

g) In the event that the required holding time to extraction or holding time to analysis was missed, flag the associated results with an "H."

h) The “X” qualifier is used only to denote the existence of presumptive evidence suggesting that the reported analyte is not present in the sample. That is, this qualifier may be used only to indicate that the chemist believes the result to be a false positive. When the “X” qualifier is used, laboratories must provide supporting data and explanatory case narrative comments in the data package.

i) Analytical results associated with a matrix spike duplicate analysis that was outside the control limit shall be qualified with a “*” flag.

j) When a sample is re-extracted and re-analyzed and both the original results and re-extracted results are reported (e.g., when surrogates fail but the re-extraction was outside the holding time), the sample number for the re-extracted results shall be uniquely identified using a re-extraction suffix.

4.1.12 Completeness

Partial deliverables shall not be submitted to the SDR unless specifically requested. In addition to the deliverable requirements given in this section, the SDR reserves the right to request run logs and chromatograms (organic chemistry only) relevant to samples from other laboratory clients that were run before or during the analytical run for Sandia samples. This is sometimes necessary to investigate suspected carryover contamination. Laboratories that fail to submit complete responses to such requests in a timely manner will be considered unresponsive and may be suspended from the laboratory analysis program. Further, the chromatograms submitted under this SOW provision shall not be edited or altered in any way, other than to delete client-specific information, prior to submission to the SNL/SMO.

4.1.13 Significant figures

a) A maximum of three significant figures shall be used to report the final analytical result.

b) Uncertainty and detection limit values shall be reported to no more than two significant figures.

c) Analytical results, uncertainties, and detection limits may be reported to one place beyond the last significant figure given for the MDLs, PQLs, L_c, or MDAs in the attachments. For example, the MDL for antimony in water is 0.06 mg/L. A result of 0.063 mg/L may be reported, while 0.0633 mg/L would be rounded down to 0.063 mg/L.

4.1.14 Chromatographic data presentation guidelines

When presenting chromatographic data, peak integrations must be displayed with a scale that provides sufficient detail to clearly view the integration. This is particularly important when small peaks are integrated in the presence of larger peaks, a situation that skews the display scale. As guidance, if the laboratory must enlarge the scale of the chromatogram to review the integration, then the enlarged view should be present in the data package. Particular attention should be applied to those instances where a value is observed above the MDL but below the PQL, as integration errors are prevalent in these situations and may result a false positive result.

4.2 Analytical data deliverable deadlines

4.2.1 Turn around times

a) A report of analytical results is due to the SDR 30 calendar days from the date of receipt of the last sample associated with each delivery order when standard turnaround time is requested.

Turnaround times for accelerated delivery requests shall be

- 72 hours (or less as negotiated by the SDR and the contractor),
- 7 calendar days,
- 15 calendar days,
- Turnaround times shall be mutually agreed upon by the contractor and the SNL/SMO. Reports with accelerated turnaround times shall be faxed or sent as PDF electronic files to the SNL/SMO, in the laboratory’s LIMS format if desired, with the full deliverable

due 15 days from receipt of samples.

- o Times are measured from the time samples are received at the laboratory to the time the SDR receives the data deliverable.

b) Reports for any requested reanalyses are due 10 working days from the date of the request unless required ingrowth times preclude this. In that case, the reanalysis reports are due no later than 15 working days from the request date. The SDR reserves the right to request expedited reanalyses when circumstances require this. Reimbursement shall be made according to the specifications of Section 1.6.2 of this SOW and will be at the standard turnaround time rates unless expedited reanalyses are requested. For reanalysis turnaround times less than 5 working days, payment will be at the applicable rate for the corresponding expedited analyses (subject to the stipulations of Section 1.6.2). Reanalysis reports shall be submitted according to the guidelines for the report level originally requested for that delivery order.

c) When report corrections are requested the laboratory must bear in mind that timely delivery is crucial from the perspective of completing data validation and making the data available to users. Simple corrections to reports, such as replacing incorrect pages or supplying omitted pages, must be delivered within no more than 24 hours. Corrections that require regeneration of data, or reassembly of the data package, are due within no more than 72 hours.

d) All correction submittals, both PDF and hard copy, must include a cover sheet that describes the deliverable to which they apply, what corrections are represented, and why the corrections were necessary.

e) The laboratory shall provide a proforma billing statement for each sample delivery group. The statement must clearly describe the number of samples, the analysis requested for each sample, unit cost, and estimated extended cost for each sample. The proforma must clearly reference the SNL ARCOC number(s) and be received by the SDR within 7 calendar days of sample receipt.

f) Invoices shall be submitted monthly on a single invoice for the sample delivery groups reported in that period. Invoices shall contain the SNL ARCOC numbers, associated sample delivery group numbers, and total cost. Proforma statements with detailed information should be attached to the invoice. A summary of attached proformas shall be included on the invoice cover page. Invoices shall be itemized and organized in such a way as to facilitate detailed review and cost verification without additional laboratory input. Costs for SDGs with unresolved problems as identified by the SDR staff shall not be invoiced until the problem is resolved. All invoices for a particular fiscal year (FY) shall be received by SNL by November 15th of the following FY. All data deliverable and billing problems shall be resolved by January 1st of the following FY. Sandia reserves the right to reject payment on late invoices due to failure to meet fiscal yearend deadlines.

4.2.2 Level D report deliverable deadlines

In specific instances, Level D deliverables may be needed after Level C reports were requested and received. The laboratory must be prepared to deliver additional records at a future time.

a) Level D documentation shall be maintained at the laboratory unless it is specifically requested for delivery to the SNL/SMO.

b) When Level D deliverables are requested to support data that have been delivered previously, the Level D deliverable shall be due 2 weeks from the date of the request.

c) The charge for the preparation of formal Level D data packages shall be specified in the itemized price list submitted by the laboratory.

4.2.3 Price reduction

a) All deliverables shall be due at the specified time unless express permission to deviate from the deliverable schedule is given by the SNL/SMO. Price reductions may be imposed for late deliverables at the discretion of the SNL/SMO, depending on the contributing circumstances, at the rate of 2 percent per working day for normal 30-day requests and 2 percent per calendar day for rush requests.

b) Unit prices will be those for the period when the deliverable arrives. However, the percent price reductions will be calculated based upon the originally requested turnaround time. That is, a report for results with a 7-day requested turn around that arrives on the 15th day will be paid for at the 15-day turn around rates less 16 percent.

c) Price reductions will not accumulate on weekends or holidays recognized by Sandia for normal 30-day requests.

d) Price reductions will be applied to particular parameters or analyses when data quality is reduced by failure to comply with the requirements of this SOW for those parameters or analyses. Sandia reserves the right to reject payment for analysis performed by the laboratory when the method-specified holding time was missed due to laboratory error. Unusable data resulting from noncompliance will not be paid for.

e) The NNSA Service Center AMP guidelines for reduction in payment are provided as Attachment 8.

4.2.4 Reporting Performance Evaluation results

The reports for PE samples submitted by the SDR shall be due as requested on the ARCOC; typically, 15, or 30 days from the date of sample receipt.

4.2.5 Reporting results for more than one analytical category

Level C reports that contain data for any combination of the major analytical categories (general inorganic, organic, radiochemistry, asbestos, or geotechnical) shall be organized by category. That is, the results forms, custody documents, and QC reports for each category shall be placed together in the deliverables. When Level D deliverables are requested, a separate deliverable shall be prepared for each analytical category unless the delivery of consolidated packages is negotiated in advance with the SNL/SMO.

5.0 LABORATORY HEALTH AND SAFETY, WASTE MANAGEMENT, AND ETHICS AGREEMENT REQUIREMENTS

The laboratory shall have the documents listed below, as applicable, and demonstrate their implementation through maintenance of employee training records.

- A chemical hygiene plan.
- A waste management plan.
- A radiological safety plan. The radiological safety plan, or a Sandia site-specific plan, shall require radiation screening during the sample receipt/login process for all samples submitted for chemical analysis.
- Ethics agreements. Laboratories shall have signed ethics agreements on file for all personnel contributing to project management, sample management, analysis, data review, and data reporting.

6.0 REFERENCES

U.S. Environmental Protection Agency, Methods for the Determination of Metals in Environmental Samples, EPA 600 4-91-010, June 1991, and Supplement 1, 1995.

U.S. Environmental Protection Agency, Contract Laboratory Program Statement of Work for Inorganic Analysis, Multi-media, Multi-Concentration, ILMO5.3, March 2004.

U.S. Environmental Protection Agency, Contract Laboratory Program Statement of Work for Organics Analysis, Multi-media, Multi-Concentration, OLMO4.2, May 1999.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Report SW-846, Third Edition, November 1986 (and updates).

U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA 600 4-79-020, December 1984.

U.S. Environmental Protection Agency, Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA/600/4-80/032, August 1980.

U.S. Environmental Protection Agency, Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, December 1988, Revised July 1991.

Occupational Safety and Health Administration, OSHA Analytical Methods Manual, Second Edition, Part 1, Volumes 1, 2, and 3, January 1990.

Occupational Safety and Health Administration, OSHA Analytical Methods Manual, Second Edition, Part 1, Volume 4, October 1993.

Occupational Safety and Health Administration, OSHA Analytical Methods Manual, Second Edition, Part 2, Volumes 1 and 2, August 1991.

National Environmental Laboratory Accreditation Conference, NELAC Quality Systems Standard, 2003.

Department of Energy, DOE Quality Systems for Analytical Services, Revision 2.4, October 2008.

7.0 ACRONYMS

AA	Atomic Absorption
AIHA	American Industrial Hygiene Association
AMP	Analytical Management Program
APHA	American Public Health Association
ARCOC	Analysis Request and Chain of Custody
ASTM	American Society for Testing and Materials
BOD	Biological Oxygen Demand
CAR	Corrective Action Report
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CF	Calibration Factor
CFR	Code of Federal Regulations
COC	Chain of Custody
CRA	Reporting Limit Verification for AA Methods
CRI	Reporting Limit Verification for ICP-AES and ICP-MS Methods
CU	Counting Uncertainty
DI	Deionized
DOE	U.S. Department of Energy
DOECAP	DOE Consolidated Audit Program
DOELAP	DOE Laboratory Accreditation Program
DQO	Data Quality Objective
DRO	Diesel Range Organics
EDD	Electronic Data Deliverables
EICP	Extracted Ion Current Profile
EPA	U.S. Environmental Protection Agency
FY	Fiscal Year
GALP	Good Automated Laboratory Practices
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace atomic Absorption
GFPC	Gas Flow Proportional Counter
GRO	Gasoline Range Organics
HE	High Explosives
HF	Hydrofluoric Acid
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
ICS	Interference Check Sample
ICV	Initial Calibration Verification

ID	Identification or Identifier
KPA	Kinetic Phosphorescence Analysis
Lc	Critical Level
LC/MS/MS	Liquid Chromatography/Mass Spectrometry/Mass Spectrometry
LCS	Laboratory Control Samples
LIMS	Laboratory Information Management System
LN2	Liquid Nitrogen
LQAP	Laboratory Quality Assurance Plan
MDA	Minimum Detection Activity
MDC	Minimum Detectable Concentration
MDL	Method Detection Limit
MDLV	Method Detection Limit Verification
MB	Method Blank
MS	Matrix Spike
MSD	Matrix Spike Duplicate
ND	Not Detected / Non-Detect
NELAC	National Environmental Laboratory Accreditation Conference
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute of Standards and Technology
NNSA	National Nuclear Service Administration
OSHA	Occupational Safety and Health Administration
PAT	Proficiency Analytical Testing
PB	Preparation Blank
PCB	Polychlorinated Biphenyl
PDS	Post Digestion Spike
PE	Performance Evaluation
PQL	Practical Quantitation Limit
PT	Performance Testing
QA	Quality Assurance
QC	Quality Control
QPR	Quarterly Progress Report
RER	Replicate Error Ratio
RF	Response Factor
RL	Reporting Limit
RLV	Reporting Limit Verification
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
Sandia	Sandia Corporation
SDG	Sample Delivery Group
SDR	Sandia Delegated Representative
SNL	Sandia National Laboratories

SNL/SMO	Sandia National Laboratories/Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SPE	Solid Phase Extraction
SPLP	Synthetic Precipitation Leaching Procedure
SVOC	Semi-volatile Organic Compound
TIC	Tentatively Identified Compound
TCLP	Toxicity Characterization Leaching Procedure
TOC	Total Organic Carbon
TOX	Total Organic Halides
TPU	Total Propagated Uncertainty
VOC	Volatile Organic Compound

Measurements and Symbols

°C	degrees Centigrade
keV	Kilo Electron Volt
MΩ·cm	mega ohms per centimeter
µmho/cm	micro ohm per centimeter
g	gram
m	milli (1/1000)
L	Liter
ppb	parts per billion
ppm	parts per million

GLOSSARY

ASTM Type I, II Water	For the purposes of this SOW, water types are those discussed in ASTM standard D 1193-77. ASTM Type I water has conductivity less than 0.06 μmho/cm or resistivity greater than 16.67 MΩ·cm. ASTM Type II water has conductivity less than 1 μmho/cm or resistivity greater than 1 MΩ·cm.
Chemical Analysis	A term that refers to all general inorganic, organic, and radiochemical analyses. The term "chemical analysis laboratory" refers to any laboratory performing those analyses under this SOW.
Controlled Document	A document that is subject to special preparation, distribution, and tracking protocols. The document control protocols ensure that persons in possession of documents are known, so that complete incorporation of revisions or implementation of new versions can be verified against the list of document holders.
Daily Requirements	Requirements for checking refrigerators, balances, and the like; these requirements apply only to business days. Daily requirements for instrument calibration and standards preparation refer only to days when the instruments are used.
Deliverable Levels	Specifications for classes of analytical data reports.
Delivery Order	A specific request for analysis of a sample or samples under an existing contract that provides all applicable specifications. No technical specifications are included with a delivery order except when special conditions occur.
Duplicate	A sample split taken by the sampling team and submitted as a sample for the purpose of assessing both sampling and analytical precision.
EDD	The acronym for electronic data deliverable. This is the computer file containing analytical results and associated information.
Intermediate Dilution	A dilution of some stock solution that requires further dilution before use in instrument calibration or QC sample preparation. Intermediate dilutions are not used to calibrate instruments in undiluted form.
L_c	The acronym for critical level corrected for concentration. When calculated according to the equation in the SOW, the L_c, gives the level at which there is a five percent probability or reporting a false positive for a sample containing no analyte. It's calculated sample specifically using variable values from the actual analytical conditions. L_c shall be reported for all radiochemistry results.
MDA	The acronym for minimum detection amount. The MDA provides sample-specific information about analytical measurement sensitivity in radiochemistry.

GLOSSARY

MDL	The acronym for method detection limit. This is a measure of instrument sensitivity using solutions that have been subjected to all sample preparation steps for the method. Reagent contributions to the signal are thus included in the MDL.
Optional Compounds	Compounds that are not routinely required in instrument calibration or the reporting of analytical results.
PE (or PT) Samples	Samples with known constituent concentrations that are periodically submitted to test laboratory analytical and reporting performance. These samples are not submitted as blinds in each sample shipment.
PQL	The acronym for practical quantitation limit. The PQL is defined to be 5 times the MDL under this SOW.
Reagent	A chemical of known purity that is used in analytical methods. This term does not apply to materials used to calibrate instruments or to perform QC activities. Such materials are called standards.
Record	The term applied to information that is subject to special handling requirements. In this SOW, "record" means information that must be maintained in such a way as to ensure that it can be retrieved in its entirety on demand.
Replicate	A sample split taken by the laboratory and prepared separately from the original sample for the purpose of assessing analytical precision.
SMO	The entity within Sandia that is responsible for writing technical and QA specifications for analytical chemistry, technical administration of laboratory contracts, sample shipment and tracking, and the various data verification, validation, and management functions.
SOP	The acronym for standard operating procedure. SOPs are documents prepared by a laboratory as controlled documents to describe the implementation of analytical methods in that laboratory. SOPs are also used to formally describe activities in the laboratory other than analytical processes.
Standard	Any material intended for use, possibly as a dilution, in instrument calibration or to perform QC activities.
Stock Solution	A high-concentration standard. Stock solutions are not used to calibrate instruments or as QC samples, but rather are diluted to produce the standards used to calibrate or prepare QC samples.

GLOSSARY

Working Standards	Standards used to calibrate instruments.
Worksheet	A term that refers to any form used to describe the work in a particular analytical batch. Worksheets may present the data acquired or be a cover sheet for those data.
Worksheet Review	A process for assessing the degree of compliance with laboratory and client requirements in the analysis documentation.

Attachment 1
General Inorganic Parameters and Target MDLs, PQLs

Table I. Metal Target Analytes and Required MDLs

	MDL (Method 6010/6020)		
Analyte	**Water mg/L**	**Solid[1] mg/kg**	**Air Filter µg/sample**
		Vegetation mg/kg	
Aluminum	0.05	3	10
Antimony	0.005	1	1
Arsenic	0.001	0.2	2
Barium	0.05	1	10
Beryllium	0.001	0.05	1
Boron	0.03	1	NA
Cadmium	0.001	0.1	1
Calcium	0.05	50	NA
Chromium	0.002	0.2	2
Cobalt	0.003	0.2	1
Copper	0.004	0.2	1
Iron	0.03	0.4	4
Lithium	0.01	1	NA
Lead	0.003	0.05	2
Magnesium	0.01	5	10
Manganese	0.002	0.2	2
Mercury (245.1, 7470A, 7471A)	0.0002	0.0002	0.005
Molybdenum	0.01	1	1
Nickel	0.01	0.07	1
Potassium	0.05	50	10
Selenium	0.002	0.1	2
Silver	0.007	0.2	1
Silica	0.05	1.5	200
Sodium	0.05	50	100
Strontium	0.003	0.1	10
Thallium	0.001	0.1	1
Tin	0.02	1.0	1
Titanium	0.02	1.0	NA
Uranium	0.001	0.01	1
Vanadium	0.005	1	1
Zinc	0.005	0.3	2

[1] Note: The solid MDLs listed require 2-gram samples for method 3050 digestions. For microwave digestions multiply the listed MDLs by 2. For hot plate digestions, multiply the listed MDLs by 4.

Attachment 1
General Inorganic Parameters and Target MDLs, PQLs

Table II. Miscellaneous General Inorganic Target Analytes, Methods, and Required PQLs

Analyte	Method Nos.	PQL	
		Water mg/L	**Solid mg/kg**
Acidity as $CaCO_3$	SM 2310B	10	NA
Alkalinity as $CaCO_3$	2320B	10	NA
Ammonium as N	350	0.1	2
Bicarbonate/carbonate	2320B	10	NA
Biological oxygen demand (BOD), 5 day	5210B	2	NA
Bromide	300	0.1	2
Carbon, dissolved organic (DOC)	5310	1	NA
Carbon, total organic (TOC)	5310, 9060	0.1	100
Chemical oxygen demand (COD)	410	5	NA
Chloride	300, 4500	1	20
Chlorine, Residual	4500	1	20
Chromium (VI)	218, 7196A	0.01	0.5
Color (color units)	2120	1	NA
Corrosivity (mm/year)	1110	NA	NA
Cyanide, Amenable	4500, 9010B, 9014	0.01	0.2
Cyanide, Reactive	Chapter 7, SW-846	0.01	0.2
Cyanide, total	4500, 9010B, 9014	0.01	0.2
Dissolved oxygen (DO)	4500	0.05	NA
Fluoride	300, 4500	0.1	2
Hardness as $CaCO_3$	2340	10	NA
Ignitability (°C)	1010, 1020A, 1030	1	1
Iodide	345	0.5	10
Nitrate as N	300, 353, 9210	0.1	2
Nitrate + nitrite as N	300, 353	0.1	2
Nitrite as N	300, 354	0.1	2
Oil and grease	1664, 9070, 9071A	2	100
Perchlorate (MDL)	314, 9058, LC/MS/MS	0.004	0.01
pH (unitless)	4500, 9040B	0.1	0.1
Phenols, total recoverable	420, 9065, 9066	0.005	1
o-Phosphate as P	300, 365	0.1	2
Phosphorus, total as P	365	0.01	0.2
Solids, settleable (mL/L/hr.)	2540	0.2	NA
Solids, total (TS)	2540	10	NA
Solids, total dissolved (TDS)	2540	10	NA
Solids, total suspended (TSS)	2540	3	NA
Solids, volatile	160	20	NA
Specific Conductance (μmho/cm)	120, 9050	1	NA
Sulfate	300, 375	1	20

Attachment 1
General Inorganic Parameters and Target MDLs, PQLs

Analyte	Method Nos.	PQL	
		Water	Solid
		mg/L	mg/kg
Sulfide	4500, 9030B, 9031	1	20
Sulfide, Reactive	Chapter 7, SW-846	1	20
Sulfur, Total	6010	0.1	1
Total organic halide (TOX)	9020B	0.03	NA
Total Kjeldahl nitrogen	351	0.5	10
Total petroleum hydrocarbons	418, 1664, 8440	1	20
Turbidity (NTU)	180	0.05	NA

Attachment 2
Radiochemical Parameters and Contract Required L_c, MDAs

Table I. Required Gamma Spectroscopy Radionuclides and MDAs by Matrix. Additional radionuclides may be requested for special projects.

	MDA				
Radionuclide	**Water pCi/L**	**Solid pCi/g**	**Air Filter pCi/sample**	**Urine pCi/L**	**Vegetation pCi/g**
^{7}Be	TBD	TBD	TBD	TBD	TBD
^{40}K	TBD	TBD	TBD	TBD	TBD
^{60}Co	10	0.03	20	20	0.03
^{137}Cs	10	0.04	20	20	0.04
^{22}Na	TBD	TBD	TBD	TBD	TBD
^{237}Np	TBD	TBD	TBD	TBD	TBD
^{223}Ra& ^{227}Th	TBD	TBD	TBD	TBD	TBD
^{224}Ra (^{212}Pb,^{212}Bi)	TBD	TBD	TBD	TBD	TBD
^{226}Ra (^{214}Pb,^{214}Bi)	TBD	TBD	TBD	TBD	TBD
^{228}Ra (^{228}Ac)	TBD	TBD	TBD	TBD	TBD
^{235}U (^{231}Th)	TBD	TBD	TBD	TBD	TBD
^{238}U (^{234}Th)	TBD	TBD	TBD	TBD	TBD
^{235}U	TBD	TBD	TBD	TBD	TBD
^{241}Am	30	0.3	25	20	0.5

Attachment 2
Radiochemical Parameters and Contract Required L_c, MDAs

Table II. Alpha Spectrometry Radionuclides and Required Critical Levels (L_c) by Matrix

Radionuclide	L_c (Concentration Corrected)					
	Water pCi/L	Solid pCi/g	Air Filter pCi/sample	Urine pCi/L	Vegetation pCi/g	Feces Ash pCi/g
^{241}Am	0.05	0.03	0.01	0.05	0.01	0.04
^{244}Cm	0.05	0.03	0.01	0.05	0.01	0.04
^{237}Np	0.05	0.03	0.01	0.05	0.01	0.04
^{210}Po	0.5	0.5	0.1	0.1	0.01	0.04
^{238}Pu	0.06	0.03	0.01	0.05	0.01	0.04
^{239}Pu	0.06	0.03	0.01	0.05	0.01	0.04
^{226}Ra	0.5	0.2	0.5	0.5	0.2	0.2
^{228}Th	0.08	0.04	0.01	0.05	0.01	0.04
^{230}Th	0.08	0.04	0.01	0.05	0.01	0.04
^{232}Th	0.08	0.04	0.01	0.05	0.01	0.04
^{234}U	0.1	0.1	0.02	0.05	0.01	0.05
^{235}U	0.1	0.1	0.02	0.05	0.01	0.05
^{238}U	0.1	0.1	0.02	0.05	0.01	0.05

Table III. Gas Proportional Counting Radionuclides and Required Critical Levels (L_c) by Matrix

Radionuclide	L_c (Concentration Corrected)			
	Water pCi/L	Solid pCi/g	Air Filter pCi/sample	Vegetation pCi/g
Gross α	1	1	1	1
Gross β	1	1	1	1
^{89}Sr	1	5	2	5
^{90}Sr	1	0.5	2	5
^{131}I	2	5	5	5
^{210}Pb	1	5	2	5
^{210}Po	1	1	2	1
^{226}Ra	1	1	2	1
^{228}Ra	0.5	0.5	1	0.5
^{99}Tc	1	5	2	5

Attachment 2
Radiochemical Parameters and Contract Required L_c, MDAs

Table IV. Liquid Scintillation Counting Radionuclides and Required Critical Levels (L_c) by Matrix

Radionuclide	L_c (Concentration Corrected) Water pCi/L	Solid/Soil pCi/g	Air Filter pCi/sample	Swipe pCi/100cm^2
^{3}H	200*	200^{1*} (pCi/L)	10	10
^{14}C	500	10	20	20
^{99}Tc	1	2	20	20
^{210}Pb	1.0	5	10	10
^{222}Rn	200	200		

[1] For tritium the specified solid L_c applies to the distilled water. For waste samples the required L_c is 0.01 pCi/g. *For tritium the L_c of the distilled water pulled from environmental soil or groundwater.

Attachment 3
Organic Parameters and Target MDLs

Table I. Target MDLs for Petroleum Hydrocarbons

Method Modification/Analyte	Water MDL, µg/L	Solid MDL, mg/kg
Gasoline Range Organics (GRO)	10	1
Diesel Range Organics (DRO)	10	1
Total Petroleum Hydrocarbons (TPH)	10	1

Table II. Organochlorine Pesticides Target Analyte List and MDLs

Compound Name	CAS No.	Water MDL, µg/L	Solid MDL, µg/kg
Aldrin	309-00-2	0.004	0.14
α-BHC	319-84-6	0.003	0.10
β-BHC	319-85-7	0.006	0.20
δ-BHC	319-86-8	0.009	0.30
γ-BHC (Lindane)	58-89-9	0.004	0.14
α-Chlordane	5103-71-9		
γ-Chlordane	5103-74-2		
4,4'-DDD	72-54-8	0.012	0.40
4,4'-DDE	72-55-9	0.012	0.41
4,4'-DDT	50-29-3	0.012	0.41
Dieldrin	60-57-1	0.002	0.07
Endosulfan I	959-98-8	0.014	0.48
Endosulfan II	33213-65-9	0.004	0.14
Endosulfan sulfate	1031-07-8	0.066	2.2
Endrin	72-20-8	0.006	0.20
Endrin aldehyde	7421-93-4	0.023	0.78
Endrin ketone	53494-70-5		
Heptachlor	76-44-8		
Heptachlor epoxide	1024-57-3	0.083	2.8
4,4’-Methoxychlor	72-43-5		
Toxaphene	8001-35-2	0.24	8.2

Attachment 3
Organic Parameters and Target MDLs

Table III. Polychlorinated Biphenyls (PCBs) Target Analyte List and MDLs

Compound Name	CAS No.	Water MDL, μg/L	Solid MDL, μg/kg
Aroclor - 1016	12674-11-2	1	33
Aroclor - 1221	11104-28-2	2	67
Aroclor - 1232	11141-16-5	1	33
Aroclor - 1242	53469-21-9	1	33
Aroclor - 1248	12672-29-6	1	33
Aroclor - 1254	11097-69-1	1	33
Aroclor - 1260	11096-82-5	1	33

Table IV. Chlorinated Herbicides Target Analyte List and MDLs

Compound Name	CAS No.	Water MDL, μg/L	Solid MDL, μg/kg
Acifluorfen	50594-66-6	0.096	3.2
Betazon	25057-89-0	0.2	6.6
Chloramben	133-90-4	0.093	3.1
2,4-D	94-75-7	0.2	6.6
2,4-DB	94-82-6	0.8	26
Dalapon	75-99-0	1.3	43
Dicamba	1918-00-9	0.081	2.7
3,5-Dichlorobenzonic acid	51-36-5	0.061	2.0
Dichlorprop	120-36-5	0.26	8.6
Dinoseb	88-85-7	0.19	6.3
MCPA	94-74-6		
MCPP	93-65-2		
4-Nitrophenol	100-02-1	0.13	4.3
Pentachlorophenol	87-86-5	0.076	2.5
Picloram	1918-02-1	0.14	4.6
2,4,5-T	93-76-5	0.08	2.6
2,4,5-TP (Silvex)	93-72-1	0.075	2.5

Attachment 3
Organic Parameters and Target MDLs

Table V. VOC Method Target Analyte List and MDLs in µg/L for Water Samples and µg/kg for Low-Level Solid Samples

Compound Name	CAS No.	25 ml purge MDL µg/L	5 gm purge MDL µg/kg
Acetone	67-64-1		
Acetonitrile	75-05-8		
Acrolein	107-02-8		
Acrylonitrile	107-13-1		
Allyl chloride	107-05-1		
Benzene	71-43-2	0.2	0.2
Bromobenzene	108-86-1	0.2	0.2
Bromochloromethane	74-97-5	0.2	0.2
Bromodichloromethane	75-27-4	0.2	0.4
Bromoform	75-25-2	0.2	0.6
Bromomethane	74-83-9	0.2	0.55
2-Butanone	78-93-3		
n-Butylbenzene	104-51-8	0.2	0.55
sec-Butylbenzene	135-98-8	0.2	0.65
tert-Butylbenzene	98-06-6	0.2	0.7
Carbon disulfide	75-15-0		
Carbon tetrachloride	56-23-5	0.2	1.05
Chloral hydrate	75-87-6		
Chlorobenzene	108-90-7	0.2	0.2
Chloroethane	75-00-3	0.2	0.5
Chloroform	67-66-3	0.2	0.2
Chloromethane	74-87-3	0.2	0.65
Chloroprene	126-99-8		
2-Chlorotoluene	95-49-8	0.2	0.2
4-Chlorotoluene	106-43-4	0.2	0.3

Attachment 3
Organic Parameters and Target MDLs

Table V. (continued) VOC Method Target Analyte List and MDLs in µg/L for Water Samples and µg/kg for Low-Level Solid Samples

Compound Name	CAS No.	25 ml purge MDL µg/L	5 gm purge MDL µg/kg
Dibromochloromethane	124-48-1	0.2	0.25
1,2-Dibromo-3-chloropropane	96-12-8	0.2	1.3
1,2-Dibromoethane	106-93-4	0.2	0.3
Dibromomethane	74-95-3	0.2	1.2
1,2-Dichlorobenzene	95-50-1	0.2	0.2
1,3-Dichlorobenzene	541-73-1	0.2	0.6
1,4-Dichlorobenzene	106-46-7	0.2	0.2
Dichlorodifluoromethane	75-71-8	0.2	0.5
1,1-Dichloroethane	75-34-3	0.2	0.2
1,2-Dichloroethane	107-06-2	0.2	0.3
1,1-Dichloroethene	75-35-4	0.2	0.6
cis-1,2-Dichloroethene	156-59-2	0.2	0.6
trans -1,2-Dichloroethene	156-60-5	0.2	0.3
1,2-Dichloroethene (total)	540-59-0		
1,2-Dichloropropane	78-87-5	0.2	0.2
1,3-Dichloropropane	142-28-9	0.2	0.2
2,2-Dichloropropane	594-20-7	0.5	1.8
1,1-Dichloropropene	563-58-6	0.2	0.5
cis-1,3-Dichloropropene	10061-01-5		
trans-1,3-Dichloropropene	10061-02-6		
1,4-Dioxane	123-91-1		
Ethylbenzene	100-41-4	0.2	0.3
Ethyl methacrylate	97-63-2		
Hexachlorobutadiene	87-68-3	0.2	0.55
2-Hexanone	591-78-6		

Attachment 3
Organic Parameters and Target MDLs

Table V. (concluded) VOC Method Target Analyte List and MDLs in µg/L for Water Samples and µg/kg for Low-Level Solid Samples

Compound Name	CAS No.	25 ml purge MDL µg/L	5 gm purge MDL µg/kg
Iodomethane	74-88-4		
Isobutyl alcohol	78-83-1		
Methacrylonitrile	126-98-7		
Methyl methacrylate	80-62-6		
Isopropylbenzene	98-82-8	0.2	0.75
4-Isopropyltoluene	99-87-6	0.2	0.6
Methylene chloride	75-09-2	0.2	0.2
4-Methyl-2-pentanone	108-10-1		
Naphthalene	91-20-3	0.2	0.2
2-Picoline	109-06-8		
Propionitrile	107-12-0		
n-Propylbenzene	103-65-1	0.2	0.2
Styrene	100-42-5	0.2	0.2
1,1,1,2-Tetrachloroethane	630-20-6	0.2	0.25
1,1,2,2-Tetrachloroethane	79-34-5	0.2	0.2
Tetrachloroethene	127-18-4	0.2	0.7
Toluene	108-88-3	0.2	0.55
1,2,3-Trichlorobenzene	87-61-6	0.2	0.2
1,2,4-Trichlorobenzene	120-82-1	0.2	0.2
1,1,1-Trichloroethane	71-55-6	0.2	0.4
1,1,2-Trichloroethane	79-00-5	0.2	0.5
Trichloroethene	79-01-6	0.2	0.95
Trichlorofluoromethane	75-69-4	0.2	0.4
1,2,3-Trichloropropane	96-18-4	0.5	1.6
1,2,4-Trimethylbenzene	95-63-6	0.2	0.65
1,3,5-Trimethylbenzene	108-67-8	0.2	0.65
Vinyl actetate	108-05-4		
Vinyl chloride	75-01-4	0.2	0.85
o-Xylene	95-47-6	0.2	0.55
m-Xylene	108-38-3	0.2	0.25
p-Xylene	106-42-3	0.2	0.65
Xylenes (total)	1330-20-7		

Attachment 3
Organic Parameters and Target MDLs

Table VI. SVOC Method Target Analyte List and MDLs in μg/L for Water Samples and μg/kg for Solid Samples

Compound Name	CAS No.	MDLs	
		Water μg/L	**Solid μg/kg**
Acenaphthene	83-32-9	5	100
Acenaphthylene	208-96-8	5	120
Acetophenone	98-86-2		
2-Acetylaminofluorene	53-96-3		
Aldrin	309-00-2	5	100
4-Aminobiphenyl	92-67-1		
Aniline	62-53-3		
Anthracene	120-12-7	5	100
Aroclor - 1016	12674-11-2		
Aroclor - 1221	11104-28-2	30	990
Aroclor - 1232	11141-16-5		
Aroclor - 1242	53469-21-9		
Aroclor - 1248	12672-29-6		
Aroclor - 1254	11097-69-1	40	1200
Aroclor - 1260	11096-82-5		
Azobenzene	103-33-3		
Benzidine	92-87-5	50	1450
Benz(a)anthracene	56-55-3	10	300
Benzo(b)fluoranthene	205-99-2	5	200
Benzo(k)fluoranthene	207-08-9	5	100
Benzo(g,h,i)perylene	191- 24-2	5	1450
Benzo(a)pyrene	50-32-8	5	1000
Benzoic acid	65-85-0		
Benzyl alcohol	100-51-6		
α-BHC	319-84-6		
β-BHC	319-85-7	5	200

Attachment 3
Organic Parameters and Target MDLs

Table VI. (continued) SVOC Method Target Analyte List and MDLs in μg/L for Water Samples and μg/kg for Solid Samples

Compound Name	CAS No.	MDLs Water μg/L	 Solid μg/kg
δ-BHC	319-86-8	10	100
γ-BHC (Lindane)	58-89-9		
Bis(2-chloroethoxy) methane	111-91-1	10	200
Bis(2-chloroethyl) ether	111-44-4	10	200
Bis(2-chloroisopropyl) ether	108-60-1	10	200
Bis(2-ethylhexyl) phthalate	117-81-7	5	100
4-Bromophenyl phenyl ether	101-55-3	5	100
Butyl benzyl phthalate	85-68-7	5	100
Carbazole	86-74-8		
Chlordane	57-74-9		
4-Chloroaniline	106-47-8		
Chlorobenzilate	510-15-6		
4-Chloro-3-methylphenol	59-50-7	5	100
2-Chloronaphthalene	91-58-7	5	100
2-Chlorophenol	95-57-8	5	150
4-Chlorophenyl phenyl ether	7005-72-3	5	150
Chrysene	218-01-9	5	100
4,4'-DDD	72-54-8	5	100
4,4'-DDE	72-55-9	10	200
4,4'-DDT	50-29-3	5	200
Demeton-O	298-03-3		
Demeton-S	126-75-0		
Diallate (cis or trans)	2303-16-4		
Dibenz(a,h)anthracene	53-70-3	5	100
Dibenzofuran	132-64-9		

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Organic Parameters and Target MDLs

Table VI. (continued) SVOC Method Target Analyte List and MDLs in μg/L for Water Samples and μg/kg for Solid Samples

Compound Name	CAS No.	MDLs	
		Water μg/L	Solid μg/kg
Di-n-butyl phthalate	84-74-2		
1,2-Dichlorobenzene	95-50-1	5	100
1,3-Dichlorobenzene	541-73-1	5	100
1,4-Dichlorobenzene	106-46-7	5	200
3,3'-Dichlorobenzidine	91-94-1	20	600
2,4-Dichlorophenol	120-83-2	5	100
2,6-Dichlorophenol	87-65-0		
Dieldrin	60-57-1	5	100
Diethyl phthalate	84-66-2	5	100
Dimethoate	60-51-5		
p-Dimethylaminoazobenzene	60-11-7		
7,12-Dimethylbenz(a)anthracene	57-97-6		
3,3'-Dimethylbenzidine	119-93-7		
2, 4-Dimethylphenol	105-67-9	5	100
Dimethyl phthalate	131-11-3	5	100
1,3-Dinitrobenzene	99-65-0		
4,6-Dinitro-2-methylphenol	534-52-1	25	800
2,4-Dinitrophenol	51-28-5	50	1500
2,4-Dinitrotoluene	121-14-2	10	200
2,6-Dinitrotoluene	606-20-2	5	100
Dinoseb	88-85-7		
Diphenylamine	122-39-4		
Di-n-octyl phthalate	117-84-0	5	100
Disulfoton	298-04-4		

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Organic Parameters and Target MDLs

Table VI. (continued) SVOC Method Target Analyte List and MDLs in μg/L for Water Samples and μg/kg for Solid Samples

Compound Name	CAS No.	MDLs Water μg/L	 Solid μg/kg
Endosulfan I	959-98-8		
Endosulfan II	33213-65-9		
Endosulfan sulfate	1031-07-8	10	200
Endrin	72-20-8		
Endrin aldehyde	7421-93-4		
Ethyl methanesulfonate	62-50-0		
Famphur	52-85-7		
Fluoranthene	206-44-0	5	100
Fluorene	86-73-7	5	100
Heptachlor	76-44-8	5	100
Heptachlor epoxide	1024-57-3	5	100
Hexachlorobenzene	118-74-1	5	100
Hexachlorobutadiene	87-68-3	5	50
Hexachlorocyclopentadiene	77-47-4		
Hexachloroethane	67-72-1	5	100
Hexachlorophene	70-30-4		
Hexachloropropene	1888-71-7		
Indeno(1,2,3-cd)pyrene	193-39-5	5	200
Isodrin	465-73-6		
Isophorone	78-59-1	5	100
Isosafrole	120-58-1		
Kepone	143-50-0		
Methapyrilene	91-80-5		
Methoxychlor	72-43-5		

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Organic Parameters and Target MDLs

Table VI. (continued) SVOC Method Target Analyte List and MDLs in μg/L for Water Samples and μg/kg for Solid Samples

Compound Name	CAS No.	MDLs Water μg/L	MDLs Solid μg/kg
3-Methylcholanthrene	56-49-5		
Methyl methanesulfonate	66-27-3		
2-Methynaphthalene	91-57-6		
Methyl parathion	298-00-0		
2-Methylphenol	95-48-7		
3-Methylphenol	108-39-4		
4-Methylphenol	106-44-5		
Naphthalene	91-20-3	5	100
1,4-Naphthoquinone	130-15-4		
1-Naphthylamine	134-32-7		
2-Naphthylamine	91-59-8		
2-Nitroaniline	88-74-4		
3-Nitroaniline	99-09-2		
4-Nitroaniline	100-01-6		
Nitrobenzene	98-95-3	5	100
2-Nitrophenol	88-75-5	5	200
4-Nitrophenol	100-02-7	5	100
Nitroquinoline-1-oxide	56-57-5		
N-Nitrosodibutylamine	924-16-3		
N-Nitrosodiethylamine	55-18-5		
N-Nitrosodimethylamine	62-75-9		
N-Nitrosodiphenylamine	86-30-6	5	100
N-Nitroso-di-n-propylamine	621-64-7		
N-Nitrosomethylethylamine	10595-95-6		

Table VI. (continued) SVOC Method Target Analyte List and MDLs in μg/L for Water Samples and μg/kg for Solid Samples

		MDLs	
		Water	Solid
Compound Name	**CAS No.**	**μg/L**	**μg/kg**
N-Nitrosomorpholine	59-89-2		
N-Nitrosopiperidine	100-75-4		
N-Nitrosopyrrolidine	930-55-2		
5-Nitro-o-toluidine	99-55-8		
2,2'-Oxybis (1-chloropropane)	108-60-1		
Parathion	56-38-2		
Pentachlorobenzene	608-93-5		
Pentachloroethane	76-01-7		
Pentachloronitrobenzene	82-68-8		
Pentachlorophenol	87-86-5	5	200
Phenacetin	62-44-2		
Phenanthrene	85-01-8	10	200
Phenol	108-95-2	5	50
1,4-Phenylenediamine	106-50-3		
Phorate	298-02-2		
2-Picoline	109-06-8		
Pronamide	23950-58-5		
Pyrene	129-00-0	5	100
Pyridine	110-86-1		
Safrole	94-59-7		
Sulfotep	3689-24-5		
1,2,4,5-Tetrachlorobenzene	95-94-3		
2,3,4,6-Tetrachlorophenol	58-90-2		
Thionazine	297-97-2		

Table VI. (concluded) SVOC Method Target Analyte List and MDLs in µg/L for Water Samples and µg/kg for Solid Samples

		MDLs	
Compound Name	**CAS No.**	**Water µg/L**	**Solid µg/kg**
o-Toluidine	95-53-4		
Toxaphene	8001-35-2		
1,2,4-Trichlorobenzene	120-82-1	5	100
2,4,5-Trichlorophenol	95-95-4		
2,4,6-Trichlorophenol	88-06-2	5	100
0,0,0-Triethyl phosphorothioate	126-68-1		

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Organic Parameters and Target MDLs

Table VII. Polychlorinated Dioxins and Furans Target Analyte List and MDLs

Compound Name	CAS No.	Water MDL ng/L	Solid MDL μg/kg
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	10	1
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	40321-76-4	25	2.5
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6	25	2.5
1,2,3,6,7,8-HxCDD	57653-85-7	25	2.5
1,2,3,7,8,9-HxCDD	19408-74-3	25	2.5
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-46-9	25	2.5
1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin (OCDD)	3236-87-9	50	5
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9	10	1
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6	25	2.5
2,3,4,7,8-PeCDF	57117-31-4	25	2.5
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9	25	2.5
1,2,3,6,7,8-HxCDF	57117-44-9	25	2.5
1,2,3,7,8,9-HxCDF	72918-21-9	25	2.5
2,3,4,6,7,8-HxCDF	60851-34-5	25	2.5
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4	25	2.5
1,2,3,4,7,8,9-HpCDF	55673-89-7	25	2.5
1,2,3,4,5,6,7,8-Octachlorodibenzofuran (OCDF)	39001-02-0	50	5
Total TCDD	41903-57-5		
Total PeCDD	36088-22-9		
Total HxCDD	34465-46-8		
Total HpCDD	37871-00-4		
Total TCDF	55722-27-5		
Total PeCDF	30402-15-4		
Total HxCDF	55684-94-1		
Total HpCDF	38998-75-3		

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Organic Parameters and Target MDLs

Table VIII. Chorinated Biphenyl (CB) Congeners and EMDLs

Chlorination levels	Congener Nos.	Water EMDL, pg/L	Solid EMDL, ng/kg
MoCB (monochlorobiphenyl)	1 to 3	5	0.5
DiCB (dichlorobiphenyl)	4 to 15	5	0.5
TrCB (trichlorobiphenyl)	16 to 39	5	0.5
TeCB (tetrachlorobiphenyl)	40 to 81	5	0.5
PeCB (pentachlorobiphenyl)	82 to 127	5	0.5
HxCB (hexachlorobiphenyl)	128 to 169	5	0.5
HpCB (heptachlorobiphenyl)	170 to 193	5	0.5
OcCB (octachlorobiphenyl)	194 to 205	5	0.5
NoCB (nonachlorobiphenyl)	206 to 208	5	0.5
DeCB (decachlorobiphenyl)	209	5	0.5

Table IX. Nitroaromatics and Nitramines Target Analyte List and MDLs

Compound Name	CAS No.	Water MDL, µg/L	Solid MDL, µg/kg
2-Amino-4,6-Dinitrotoluene (2-Am-DNT)	355-72-78-2	10	250
4-Amino-2,6-Dinitrotoluene (4-Am-DNT)	1946-51-0	10	250
1,3-Dinitrobenzene (DNB)	99-65-0	10	250
2,4-Dinitrotoluene (24DNT)	121-14-2	10	250
2,6-Dinitrotoluene (26DNT)	606-20-2	5	250
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	20	1000
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8	50	500
Nitrobenzene (NB)	98-95-3	10	250
Nitroglycerine (NG)			
2-Nitrotoluene (2NT)	88-72-2	10	250
3-Nitrotoluene (3NT)	99-08-1	10	250
4-Nitrotoluene (4NT)	99-99-0	10	250
Pentaerythritol tetranitrate (PETN)			
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	20	2000
1,3,5-Trinitrobenzene (135TNB)	99-35-4	10	250
2,4,6-Trinitrotoluene (TNT)	118-96-7	10	250

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Organic Parameters and Target MDLs

Table X. VOCs in Ambient Air Target Analyte List and MDLs

Compound Name	CAS No.	MDL, ppbv
Acetone	67-54-1	
Benzene	71-43-2	0.2
Benzyl chloride	100-44-7	0.2
Bromomethane	74-83-9	0.2
2-Butanone (MEK)	78-93-3	
Carbon tetrachloride	56-23-5	0.2
Chlorobenzene	108-90-7	0.2
Chloroethane	75-00-3	0.2
Chloroform	67-66-3	0.2
Chloromethane	74-87-3	0.2
1,2-Dibromoethane	106-93-4	0.2
1,2-Dichlorobenzene	95-50-1	0.2
1,3-Dichlorobenzene	541-73-1	0.2
1,4-Dichlorobenzene	106-46-7	0.2
Dichlorodifluoromethane (Freon 12)	75-71-8	0.2
1,1-Dichloroethane	75-34-3	0.2
1,2-Dichloroethane	107-06-2	0.2
1,1-Dichloroethene	75-35-4	0.2
cis-1,2-Dichloroethene	156-59-2	0.2
1,2-Dichloropropane	78-87-5	0.2
cis-1,3-Dichloropropene	10061-01-5	0.2
trans-1,3-Dichloropropene	10061-02-6	0.2
1,2-Dichloro-1,2,2,2-tetrafluoroethane (Freon 114)	76-14-2	0.2
Ethylbenzene	100-41-4	0.2
4-Ethyltoluene	622-96-8	0.2
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	354-58-5	0.2
Hexachlorobutadiene	87-68-3	0.2
Methylene chloride	75-09-2	0.2
4-Methyl-2-pentanone (MIBK)	108-10-1	
Styrene	100-42-5	0.2
1,1,2,2-Tetrachloroethane	79-34-5	0.2
Tetrachloroethene	127-18-4	0.2
Toluene	108-88-3	0.2
1,2,4-Trichlorobenzene	120-82-1	0.2
1,1,1-Trichloroethane	71-55-6	0.2
1,1,2-Trichloroethane	79-00-5	0.2
Trichloroethene	79-01-6	0.2
Trichlorofluoromethane (Freon 11)	75-69-4	0.2
1,2,4-Trimethylbenzene	95-63-6	0.2
1,3,5-Trimethylbenzene	108-67-8	0.2
Vinyl chloride	75-01-4	0.2
o-Xylene	95-47-6	0.2
m-Xylene	108-38-3	0.2
p-Xylene	106-42-3	0.2

Attachment 3
Organic Parameters and Target MDLs

Table XI. Pharmaceutical and Personal Care Products (PPCPs) Target Analyte List and MDLs

Compound Name	CAS No.	MDL ng/L	RL ng/L
1,7-Dimethylxanthine	611-59-6	67	200
Acetaminophen	103-90-2	6.9	20
Albuterol	18559-94-9	0.47	10
Atenolol	29122-68-7	33	100
Atorvastatin	110862-48-1	100	250
Bisphenol A	80-05-7	100	150
Caffeine	58-08-2	13	50
Carbadox	6804-07-5	10	50
Carbamazepine	298-46-4	2.2	10
Cotinine	486-56-6	1.4	10
DEET	134-62-3	25	25
Diphenhydramine hydrochloride	147-24-0	3	10
Fluoxetine	54910-89-3	9.8	25
Gemfibrozil	25812-30-0	11	25
Ibuprofen	15687-27-1	6.3	25
Iopromide	73334-07-3	6.9	50
Lincomycin	154-21-2	5	10
Lorazepam	846-49-1	0.8	10
Methadone	76-99-3	1.9	10
Morphine	57-27-2	9.3	100
Naproxen	22204-53-1	17	50
Ormetoprim	6981-18-6	2.8	10
Oxolinic acid	14698-29-4	6.9	20
Phenytoin	57-41-0	61	100
Primidone	125-33-7	100	250
Ranitidine	66357-35-5	3.6	10
Salicylic acid	69-72-7	50	50
Sucralose	56038-13-2	250	500
Sulfachloropyridazine	80-32-0	2.7	10
Sulfadiazine	68-35-9	1.1	10
Sulfadimethoxine	122-11-2	6	10
Sulfamerazine	127-79-7	5	10
Sulfamethazine	57-68-1	8.4	10
Sulfamethizole	144-82-1	3.4	10
Sulfamethoxazole	723-46-6	4.9	10
Sulfanilamide	63-74-1	200	200
Sulfathiazole	72-14-0	3.7	10
Thiabendazole	148-79-8	5.2	10
Triclocarban	101-20-2	3.3	10
Triclosan	3380-34-5	6.1	50

Table XI. (concluded) Pharmaceutical and Personal Care Products (PPCPs) Target Analyte List and MDLs

Compound Name	CAS No.	MDL ng/L	RL ng/L
Trimethoprim	738-70-5	4	10
Tris(1,3-dichloro-2-propyl)phosphate	13674-87-8	250	500
Tris(1-chloro-2-propyl)phosphate	13674-84-5	15	50
Tris(2-chloroethyl)phosphate	115-96-8	20	50
Tylosin	1401-69-0	1.1	10
Warfarin	81-81-2	6.9	20

Attachment 4
Geotechnical Methods

Table I. Geotechnical Test Methods

Method Title	ASTM No.
Atterberg Limits, Liquid Limit, and Plastic Limit	D4318
Compression Test, Unconfined Test for Rock	D2938
Consolidated-Undrained Triaxial Compression Test, Cohesive Soils	D4767
Direct Shear Test of Soils Under Consolidated Conditions	D3080
Dry Preparation of Samples for Particle-Size Analysis	D421
Laboratory Compaction Characteristics, Soil	D698
Particle-Size Analysis, Soil	D422
Preparation and Transport of Rock Samples	D5079
Preparation and Transport of Soil Samples	D4220
Specific Gravity, Soil	D854
Triaxial Compressive Strength, Rock	D2664
Water Content, Soil and Rock	D2216
Wet Preparation of Samples for Particle-Size Analysis	D2217

Note: The ASTM methods are from the 1996 Annual Books of ASTM Standards, Section 4, Construction, Volume 04.08 Soil and Rock (I) and Volume 04.09 Soil and Rock (II).

Attachment 5
Holding Times and Preservation Techniques

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times	
					Sample	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,	Total Cyanide	Water	1 L Plastic	≤6°C; NaOH; pH > 12	14 Days	NA
9014, ,	Amenable Cyanide	Solid/Other	125 mL Glass Jar	≤6°C	14 Days	NA
335.4, 4500CN-G						
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,	All metals except Cr(VI) and Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	180 Days	NA
6010B, 6020		Solid/Other	250 mL Glass Jar		180 Days	NA
3060A	Cr(VI)	Water	500 mL Plastic	≤6 °C	24 Hours	NA
218.6		Water	500 mL Plastic	≤6°C, pH 9-9.5	28 Days	NA
7197, 7196A		Solid/Other	250 mL Glass Jar	≤6 °C	30 Days	7 Days
245.1, 7470A,	Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	28 Days	NA
7471A		Solid/Other	250 mL Glass Jar	≤6 °C	28 Days	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
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						
300.0 354.1	Nitrate, Nitrite, Ortho Phosphorus	Water	500 mL Plastic	≤6 °C	48 Hours	NA
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

Attachment 5
Holding Times and Preservation Techniques

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	 Extract
314.0, 9058	Perchlorate by IC	Water	250 mL Plastic or Glass	≤6 °C	28 Days	NA
6850 (modified)	Perchlorate by LC/MS/MS	Water	250 mL Plastic or Glass	≤6 °C	28 Days	60 days
6860 (modified)						
330,0, 331.0		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
9070/9071A	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	
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
9060	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	TPH	Water	1 L Amber Glass	≤6 °C; HCl; pH < 2	28 Days	NA
1664	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

Attachment 5
Holding Times and Preservation Techniques

					Holding Times	
Method	**Parameters**	**Matrix**	**Volume/Container**	**Preservation**	**Sample**	**Extract**
9040B $4500H^+$-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, 9050	Specific Conductance	Water	125 mL Plastic	≤6 °C	ASAP	NA
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
8015 (Modified)	Petroleum Hydrocarbons (Diesel Range Organics)	Water	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	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/8015 (Modified)	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	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
8081	Organochlorine Pesticides	Water	4 L Amber Glass Bottle	≤6 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	≤6 °C	14 Days	40 Days
8082	PCBs	Water	4 L Amber Glass Bottle	≤6 °C	1 Year	1 Year
		Soil/Other	250 Glass Jar	≤6 °C	1 Year	1 Year
8141A	Organophosphorous Compounds	Water	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

Attachment 5
Holding Times and Preservation Techniques

					Holding Times	
Method	**Parameters**	**Matrix**	**Volume/Container**	**Preservation**	**Sample**	**Extract**
8151A	Chlorinated Herbicides	Water	4 L Amber Glass Bottle	≤6 °C	7 Days	40 Days
		Soil/Other	250 Glass Jar	≤6 °C	14 Days	40 Days
8260C (Modified)	Volatile Organics by GC-MS	Water^{1,2}	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/8260C	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
8270D	Semivolatile Organics by GC-MS	Water	4 L Amber Glass Bottle	≤6 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	≤6°C	14 Days	40 Days
8280A	Polychlorinated Dioxins and Furans by GC/MS	Water	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/LRMS	Water	4 L Amber Glass Bottle	≤6 °C	30 Days (1 Yr)	45 Days
		Soil/Other	250 mL Glass Jar	≤6 °C	30 Days (1 Yr)	45 Days
1613	Dioxins and Furans by Isotope Dilution HRGC/LRMS	Water	Amber Glass	≤6 °C	1 Year	1 Year
		Solid	Amber Glass Jar	≤6 °C	1 Year	1 Year
1668A	PCB Congeners by HRGC/LRMS	Water	Amber Glass	≤6 °C, H_2SO_4; pH 2-3	1 Year	1 Year
		Solid	Amber Glass Jar	≤6 °C	1 Year	1 Year
1694	PPCPs	Water^{3}	Amber Glass	≤6 °C	7 Days	30 Days
		Solid	Amber Glass Jar	≤6 °C	7 Days	30 Days
8318	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	PAHs	Water	Amber Glass/Teflon lined cap	≤6 °C	7 Days	40 Days
TO-13A	PAHs in Filter Cartridges	PUF, Tenax, or XAD-2 Filter Cartridge		≤6 °C	7 Days	40 Days
TO-14A	VOC in Air	SUMMA® Canister			28 Days (by consensus)	

Attachment 5
Holding Times and Preservation Techniques

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times Extract
8321(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

Notes:

[1] If vinyl chloride, styrene or 2-chloroethylvinylether are analytes of interest, collect a second set of samples without preservatives and analyzed within 7 days.

[2] If acrolein and acrylonitrile are analytes of interest, adjust to pH 4-5.

[3] If residual chlorine present, preserve with ascorbic acid.

Attachment 6
Target Surrogate Recovery Control Limits

Method 8081 and 8082: Organochlorine Pesticides and PCBs as Aroclors Required Surrogate Compounds

Surrogate Compounds	CAS No.	Acceptance Criteria	
		Water	Soil
Decachlorobiphenyl	2051-24-3	50-160%	50-160%
Tetrachloro-m-xylene	877-09-8	50-160%	50-160%

Method 8260C: Required Surrogate Compounds

Surrogate Compounds	CAS No.	Acceptance Criteria	
		Water	Soil
4-Bromofluorobenzene	460-00-4	86-115%	74-121%
1,2-Dichloroethane-d4	17060-07-0	80-120%	80-120%
Toluene-d8	2037-26-5	88-110%	81-117%
Dibromofluoromethane		86-118%	80-120%

Method 8270D: Required Surrogate Compounds

Surrogate Compounds	CAS No.	Acceptance Criteria	
		Water	Soil
2-Fluorobiphenyl	321-60-8	43-116%	30-115%
2-Fluorophenol	367-12-4	21-110%	25-121%
Nitrobenzene-d5	4165-60-0	35-114%	23-120%
Phenol-d6	13127-88-3	10-110%	24-113%
p-Terphenyl-d14	1718-51-0	33-141%	18-137%
2,4,6-Tribromophenol	118-79-6	10-123%	19-122%

Attachment 6
Target Surrogate Recovery Control Limits

Method 8330A: Nitroaromatics and Nitramines
Required Surrogate Compounds
(use either or both)

Surrogate Compounds	CAS No.	Acceptance Criteria*	
		Water	Soil
3,4-Dinitrotoluene	610-39-9	50-160%	50-160%
2-Methyl-4-nitroaniline	99-55-8	50-160%	50-160%
1,4-Dintrobenzene (recommended)	100-25-4	50-160%	50-160%
1,2-Dintrobenzene (recommended)	528-29-0	50-160%	50-160%

*Specific surrogates are not designated in the method.

Method 8151: Chlorinated Herbicides
Required Surrogate Compounds

Surrogate Compounds	CAS No.	Acceptance Criteria	
		Water	Soil
2,4-Dichlorophenylacetic Acid (DCAA)		50-160%	50-160%

Attachment 7
Analytical Request and Chain of Custody Form

Internal Lab:

Page _1_ of _1_

Batch No.:

SMO Use

ARCOC

Project Name:	Date Samples Shipped:	SMO Authorization.:	☐ Waste Characterization
Project/Task Manager:	Carrier/Waybill No.	SMO Contact Phone:	☐ RMMA
Project/Task Number:	Lab Contact:		☐ Released by COC No.
Service Order:	Lab Destination:	Send Report to SMO:	☐ 4° Celsius
	Contract No.:		Bill to: Sandia National Laboratories (Accounts Payable); P.O. Box 5800, MS-0154; Albuquerque, NM 87185-0154
Tech Area:			
Building : **Room:**	**Operational Site:**		

Sample Number	Fraction	Sample Location Detail	Depth (ft)	Date/Time(hr) Collected	Sample Matrix	Container		Preser-vative	Collect Method	Sample Type	Parameter & Method Requested	Lab Sample Id
						Type	Vol					

Last Chain: ☐ Yes	**Sample Tracking** **SMO Use**	**Special Instructions/QC Requirements:**	Abnormal Conditions on Receipt
Validation Req'd: ☐ Yes	Date Entered:	**EDD:** **Yes** ☐ **No** ☐	
Background: ☐ Yes	Entered by:	**Turnaround Time** ☐ _7 Day*_ ☐ 15 Day * ☐ 30 Day	
Confirmatory: ☐ Yes	QC inits.:	**Negotiated TAT:** ☐	

	Name	Signature	Init.	Company/Org/Phone/Cell
Sample Team Members				

Sample Disposal: ☐ Return to Client ☐ Disposal by Lab

Return Samples By:

Comments:

Please list as separate report.

Lab Use

1. Relinquished by	Org.	Date	Time	3. Relinquished by	Org.	Date	Time
1. Received by	Org.	Date	Time	3. Received by	Org.	Date	Time
2. Relinquished by	Org.	Date	Time	4. Relinquished by	Org.	Date	Time
2. Received by	Org.	Date	Time	4. Received by	Org.	Date	Time

***Prior confirmation with SMO required for 7 and 15 day TAT**

Attachment 8
AMP Policy for Payment Reduction
NNSA Service Center Sites

In order to ensure that adequate value is obtained for dollars spent, it is sometimes necessary to impose a payment reduction or to elect nonpayment for laboratory analytical services. At the same time, it is in the interest of the DOE to maintain good working relationships with its contract laboratories. Since payment options can be abused in the hands of inexperienced staff, senior management needs to closely monitor their use to ensure that those relationships are not needlessly damaged. Some guidelines for a payment reduction or nonpayment election are listed below.

1) If a laboratory fails to meet QC criteria, and if reanalysis is precluded by expiration of holding times, then a payment reduction or nonpayment may be appropriate.

2) If deliverables do not contain the required supporting documentation, and if the laboratory cannot deliver such documentation upon request, then the data quality is negatively affected and a payment reduction or nonpayment may be appropriate.

3) If holding times are missed, and if it can be demonstrated that the samples did arrive at the laboratory far enough in advance to allow reasonable time for the analyses, then the laboratory is responsible for reduced data quality. A payment reduction or nonpayment may be appropriate under these circumstances. However, if laboratory personnel notified the project that holding times would be missed far enough in advance to select and ship to another laboratory, then no payment reduction should be assessed. Further, if samples arrive at the laboratory very close to expiration, and if project sampling or SMO personnel did not tell the laboratory to expect this, then a payment reduction is generally inappropriate.

4) If a laboratory fails to meet deliverable schedules for analytical reports it may be necessary to impose a payment reduction. Such action should be taken only in particularly egregious or chronic cases. (Rigid contract stipulations already in place at some NNSA Service Center facilities may supersede this guideline.)

5) There are cases where a laboratory uses an unapproved analytical technique, due to catastrophic instrument failure or for some other reason, without first obtaining permission from the project. If the resulting data do not meet technical or regulatory requirements, a payment reduction or nonpayment may be appropriate.

6) If a laboratory uses an unapproved subcontract vendor to increase its capacity, any project data acquired by the unapproved vendor are subject to a payment reduction or nonpayment.

7) Failure to meet technical requirements, such as meeting detection limits, should not result in payment reduction unless the laboratory clearly made a technical error that is unrelated to the sample matrix and the error has an effect on data usability. This applies when the laboratory used an inadequate analytical technique, inappropriate wavelength, too-short count time, or excessive dilution.

8) Verified malfeasance, such as "dry-labing," should result in immediate suspension in addition to nonpayment for the analyses in question.

9) Failure to submit periodic progress reports, MDL studies, PE data, or CARs should not result in reduced payment for analysis data deliverables. Typically, laboratories are suspended pending compliance in such cases

Attachment 9
AMP Policy for Information Sharing
NNSA Service Center Sites

The NNSA Analytical Management Program (AMP) strives to improve the quality of the chemical analysis data acquired and to reduce duplication of effort by sharing performance information for subcontract laboratories. Because each project has its own set of performance criteria and areas of interest, it is necessary to establish guidelines for the assessment of this information.

There are many different issues and measurement criteria associated with laboratory performance. The purpose in sharing laboratory performance data among NNSA projects is to provide as much information as possible, as inexpensively as possible, to project personnel so that they can make informed decisions regarding the selection and use of laboratories. Therefore it will be the policy of the NNSA AMP to encourage open and timely dissemination of laboratory performance information, without imposing artificial requirements with regard to the specifics of how it must be used. However, NNSA facility personnel must bear in mind that the NNSA's (and its subcontractor's) relationships with analytical laboratories are important partnerships. It is not in NNSA's best interest to violate a laboratory's trust by frivolously publicizing isolated or trivial errors.

Performance-Related Reasons for Corrective Action or Suspension

Individual NNSA projects may request corrective action or, in extreme cases, even suspend subcontract laboratories from project chemical analysis programs for a variety of reasons. Some possible examples are listed below.

1) Failure to meet contractual obligations
    a) Failure to provide required documentation
    b) Chronic analytical quality control deficiencies
    c) Inability to meet deliverable schedules for analysis reports, periodic progress reports, MDL studies, or corrective action reports
    d) Failure to implement project quality assurance requirements

2) On-site audit or data package assessment findings
    a) Critical QA systems failure
    b) Critical technical systems failure
    c) Incorrect data reporting
    d) Inappropriate staff organization, such as conflict between QA and laboratory management duties
    e) Use of unapproved procedures without prior permission

3) Performance Evaluation Samples
    a) Very large analytical errors reported
    b) A systematic bias is indicated by all results when two or more PE samples are submitted
    c) False negative results reported
    d) Parameter is outside the acceptance interval in two consecutive PE rounds
    e) Parameter is outside the acceptance interval in more than one PE program.
    f) An analytical or reporting error is noted in an area where other problems are known to exist (chronic QC, technical, or reporting problems already identified)

4) Substantial Contamination or Incorrect Reporting
    a) It is discovered that a laboratory is contaminating facility samples through adverse ambient conditions, inadequate laboratory practices, or the residual effects of high-level samples from other clients
    b) Analyses at a laboratory are affected by incorrect integration or other method implementation errors, resulting in chronic misreporting of data
    c) Reported detection limits are too low, either for accurate integration or existing laboratory conditions, resulting in unacceptable false positive reporting rates

Non-Performance-Related Issues

In addition, there may be other (non-performance-related) reasons relating to laboratory capacity for a temporary hiatus from the routine flow of samples. Such reasons might include the sudden loss of critical laboratory staff members or over-committed laboratory facilities.

Differing Project Needs

Performance requirements in a given analytical area vary between projects, based on project-specific data quality objectives or facility contract specifications. Thus, what represents a deficiency for one project may be acceptable for another. Further, in the case for which a laboratory provides service in several areas (i.e. physical testing, radiochemistry, general inorganic, and organic chemistry), deficiencies noted in one area may not affect the services utilized in other areas. Therefore, each project must have the latitude to examine shared performance information, and take action or not, based on that project's specific needs.

Performance information may be shared among projects under the system now in place. If any project should decide to suspend a subcontract laboratory based on performance, other projects that also use that laboratory must decide how, if at all, the deficiency or deficiencies affect their work.

Attachment 9
AMP Policy for Information Sharing
NNSA Service Center Sites

Guidelines for Sharing Performance Information

With this in mind, the NNSA AMP recommends the following guidelines.

1) Information to be shared should be presented as clearly as possible, and should include the performance measurement criteria used to evaluate it for the home (original) project if applicable. This information should include any relevant quality assurance, quality control, technical, or reporting criteria.

2) Contact person(s) whose responsibility it is to transmit performance information to other NNSA facilities and/or receive transmitted information from other facilities should be designated at each project.

3) Personnel responsible for review of laboratory performance information received from other projects should be intimately familiar with all laboratory performance criteria specific to their own projects.

4) Review personnel should investigate and verify negative performance information from other projects that is applicable to their work. This will ensure that the information is completely and correctly understood before it is acted upon.

5) Follow-up information regarding resolution of deficiencies should be transmitted to other NNSA facilities as quickly as possible so that the laboratory capacity originally in question can become available again.

Attachment 10
Routine Sub-Sampling Procedure

Routine Soil Sub-sampling Procedure

General procedure:

1) Remove any twigs or rocks from the sample before drying and grinding.
2) Blend the entire sample prior to taking the first aliquot.
3) Use an aliquot of at least 20 times greater than the final required mass for the first aliquot to be sub-sampled for each preparation. For cases where the sample submitted is less than 20 times the final sample mass, use the entire sample mass and note the insufficient sample in the case narrative.
4) Use a square-shaped scoop for all sub-sampling to avoid any bias in favor of the large or small particles.

Specific procedures:

1) **SVOC type analyses (including PCBs, Pesticides, and Polynuclear Aromatic Hydrocarbons)**
   Soil samples submitted for SVOC analysis shall be prepared as follows:
   a) Using the first sample aliquot, randomly acquire five equal sub-samples using a square-shaped scoop to obtain the final sample mass.
   b) Document this sub-sampling in the hard copy data package.
   c) Proceed with the SVOC analysis.

2) **HE analysis**
   Soil samples submitted for HE analysis shall be prepared as follows:
   a) Dry the entire first sample aliquot, either by air drying or using a low temperature oven.
   b) Grind the entire first sample aliquot to pass through a 30 mesh sieve.
   c) Using the entire first sample aliquot, randomly acquire five equal sub-samples using a square-shaped scoop to obtain the final sample weight.
   d) Document this drying, grinding, and sub-sampling in the hard copy data package.
   e) Proceed with the HE analysis.

3) **Metals analysis**
   Soil samples submitted for metals analysis shall be prepared as follows:
   a) Dry the entire first sample aliquot, either by air drying or using a low temperature oven.
   b) Grind the entire first sample aliquot to pass through a 30 mesh sieve.
   c) Using the entire first sample aliquot, randomly acquire five equal sub-samples using a square-shaped scoop to obtain the final sample weight.
   d) Document this drying, grinding, and sub-sampling in the hard copy data package.

Attachment 11
SNL/SMO Electronic Deliverable Specification

Sandia National Laboratories

Albuquerque, New Mexico 87185-0729

date: August 21, 2012

to: Electronic Deliverable Specification Recipients

from: Sample Management Office
Sandia National Laboratories, MS-0729 (4142)

subject: Electronic Deliverable Specification Revision 18

Attached is the latest revision to the Electronic Deliverable Specification. Revision 18.

SANDIA NATIONAL LABORATORIES
NEW MEXICO
(SNL/NM)

SAMPLE MANAGEMENT OFFICE
ELECTRONIC DATA DELIVERABLE SPECIFICATION
DEPARTMENT 4142

08/21/2012

Attachment 11
SNL/SMO Electronic Deliverable Specification

Purpose

This specification describes the format for providing the electronic deliverable lab data for analytical laboratories to the Sandia National Laboratories New Mexico (SNL/NM) Sample Management Office (SMO).

Scope

This Electronic Data Deliverable (EDD) applies to all data delivered to the SMO from contract analytical laboratories in fulfillment of contract agreements**.**

Ownership

SNL/NM owns this Document and is responsible for all changes and corrections. Contact SMO Project Leader at (505) 844-3185 for comments.

Electronic Data Deliverable Specification

Procedure

The data shall be provided as an ASCII text file, comma delimited and with double quotations used as field qualifiers. The file will be delivered to the SMO using a CD with the PDF data-package file. Each EDD file will be comprised of one Analytical Request Chain of Custody (ARCOC). Each CD can be comprised of more than one file, only if, multiple ARCOCs were combined into one data package. The CD shall be externally labeled with the lab, date, ARCOC numbers, related Sample Delivery Group (SDG) numbers, and filename. Each CD will be accompanied by a transmittal letter that will indicate the sample delivery group, ARCOC numbers and pertinent instructions (i.e., this data is a re-submittal).

Each ARCOC will be stored in a unique file that will use the naming convention ARCOC#.snd.

Attachment 11
SNL/SMO Electronic Deliverable Specification

Field Definitions

Column **Field Name (Maximum Field Length) Description**

1) **Sample Number - Fraction - ER Sample ID or Sample Location Detail (50)** This number is obtained from the Analytical Request Chain of Custody (ARCOC) and consists of two blocks on the (ARCOC), 1) the Sample No. - Fraction block and 2) the ER Sample ID or Sample Location Detail block. These two blocks should be concatenated together in the Client Sample ID field and separated by a "/". An example is given here, followed by the rules:

Sample No. - Fraction	ER Sample ID or Sample Location Detail	Correct Field Entry for EDD
012345 - 001	TA2-BH-11.5	012345-001/TA2-BH-11.5

The sample number will be six characters long, padded with leading zeros. The fraction will be three digits long and padded with leading zeros except when Sandia directs a re-run, in which case the fraction will be three characters long with the first character being an "R" (i.e., 012345-R01/TA2-BH-11.5). This field should be populated for result identifier SA; BL requested by Sandia, MS/MSD, sample replicates REP, rad chemical tracers (RCT), or SUR for associated SNL samples**.** Labs shall not alter the sample number - fraction without prior written permission from the SMO Project Leader or designee. The ER sample ID or Sample Location Detail may be truncated if it exceeds the lab's field size.

2) **Sample Collection Date (8)** (MMDDYYYY) The date a sample was taken. This information is obtained from the ARCOC. This field will only be populated for result identifier SA or BL.
3) **ARCOC Number (6)** This number is obtained from the ARCOC. The labs shall not alter or truncate the ARCOC number without prior written permission from the SNL/SMO. This field will only be populated for result identifier SA or BL.
4) **Analysis Requested (70) The** analysis requested is obtained from the ARCOC. The labs may populate this field with their own naming conventions. This field will only be populated for result identifier SA and BL.
5) **Sample Receipt Date (8)** (MMDDYYYY) The date a sample was received by the lab. This field is required for all analyses performed on SMO provided samples. This field will only be populated for result identifier SA and BL.
6) **Sample Matrix (7)** The predominant material comprising the sample. This field must be filled for every record, and it must match Appendix B.
7) **Lab Name (4)** The abbreviated name for each lab. This field must be filled for every record. (See Appendix A)

8) **Sample Disposition/Comment (15)** The final disposition of the sample at the laboratory. This field is used to report a status to SMO when no results could be obtained for a sample. Required for all SMO samples with no analytical results and must match Appendix H. Leave blank for all records with analytical results.
9) **Sample Delivery Group (SDG) (10)** Input the internal lab identifier indicating the laboratory grouping of samples in which the sample was analyzed. This number must be unique. It is used to manage the data packages for validation and it is used to uniquely identify the ASCII file names provided to SNL. This field must be filled for every record.
10) **Lab Sample Number (15)** The internal lab identifier used to track a specific sample. This number will be identical for all analyses performed within the lab on a SMO sample. This field must be filled for every record.
11) **Sample Preparation Date (8)** (MMDDYYYY) Date of sample preparation at the laboratory. Use the latest preparation date when multiple preparation dates exist. This field is required for every record that has an analysis preparation type.
12) **Sample Preparation Type (3)** This is used to indicate if the reported result is for a sample preparation that was suspended, dissolved or total. Required for all analyses performed on SMO provided samples and must match Appendix F. This field will only be populated for result identifiers SA, BL, REP, MS, MSD. Assume the preparation type is TOTAL if it is not specified on the Chain of Custody that the sample was filtered or filtration requested at the laboratory.
13) **Sample Extraction/Digestion Method (15)** The coded identifier of the method used to perform the extraction or digestion (e.g., EPA-SW846-1311). This field will only be populated for result identifier SA, BL, REP, MS, MSD, RCT.
14) **Lab Method Code (25)** The internal laboratory method code used to obtain analysis. This field is required for every record. An appendix of expected codes for each lab will be created and checked for a match. This field must be filled for every record. (Provided by each lab)
15) **Instrument (10)** The coded identifier for the instrument used to perform the analysis. Each lab develops its own code. All records with a result must have this field populated.
16) **Analyst (25)** The name of the analyst or approving authority. This field must be filled for every record.
17) **Results Identifier (3)** SMO codes that differentiate between target analytical results, laboratory quality assurance samples, and analytical re-analysis. All other quantifiable analysis runs will be identified with a different unique result identifier. There will never be two analysis runs for the same sample that has the same result identifier. This field must be filled for every record, and it must match Appendix C.
18) **Analysis Date and Time (13)** (MMDDYYYY: HHMM) Laboratory analysis date and time. This field must be filled for every record.
19) **Residual Weight (4)** The weight of material on the planchet after evaporation. Used for radionuclide analyses only when applicable. Leave blank for all other analysis.
20) **Dilution Factor (5)** When a sample is diluted, the dilution factor shall be recorded in this field. The factor shall be recorded as 5, 2.5, 10, 100, 5000 or in a similar manner. This field must be filled for every record. The default is 1.

21) **CAS Numbers (12)** (NNNNN-NN-N) The unique number assigned to an analyte by the Chemical Abstract Service. This field is required if available.

22) **Parameter Name (70)** Input the analyzed compound name. The parameter name of "UNKNOWN" may be used only for Tentatively Identified Compound (TIC) analytes where appropriate. This field must be filled for every record.

23) **Flag TICs (1)** Input a "Y" only when the reported result is a TIC. This field will remain blank in all other cases.

24) **Retention Time (7)** For tentatively identified compounds (TICs) only, the retention time must be input. Required for all identified TICs.

25) **Result (10)** The analytical result for a chemical compound. Do not use any characters in the results including commas. Scientific notation and/or negative results may be reported for RAD only.

For non-RAD non-detected results only, use the MDL for organics and inorganics. For results without MDL use the PQL. For radiochemistry the result shall be the measured concentration, whatever is determined (+ or -). For radiochemistry do not default to the L_c or the MDA**.**

Report the actual measured concentration regardless if it is a positive or negative result. Do not report the string "< MDA".

26) **Spike Reference Identifier (15)** The lot number or lab designation of the spike material used for MS/MSD, Sur, RCT, or LCS samples. The standard ID that you trace through the laboratory and which is documented on the analyst's prep worksheets**.** This field will be left blank for samples and blanks.

27) **Spike Added (8)** The known spike concentration added for MS/MSD, RCT, or LCS. The unit of measure should be the same as the unit of measure for the result.

28) **Percent Recovery (5**) Report for MS/MSD, Sur, RCT, and LCS. Do not report negative values. If a percent is calculated and the result is negative due to insufficient spiking concentrations, record a "0" (zero) in the Percent Recovery column and flag it with an "N" for non-compatible spiking concentration.

29) **QC Control Upper Limit or Lab Sample Result Upper Limit (8)** This field designates the upper acceptable control limit for an analyte. For LCS, MS, RCT and Sur this limit should pertain to accuracy (Percent Recovery, Col. 28). For MSD and REP, this limit should pertain to bias (Relative Percent Recovery for organic and inorganic analysis, Replicate Error Ratio for radiochemistry analysis, Col. 31). This field will be populated for result identifier types LCS, RCT, MS, MSD, REP, and Sur.

30) **QC Control Lower Limit or Lab Sample Result Lower Limit (8)** This field designates the lower acceptable control limit for an analyte in the sample solution. For LCS, MS, RCT, and Sur this limit should pertain to accuracy (Percent Recovery, Col. 28). For MSD and REP this limit should pertain to bias (Relative Percent Recovery for organic and inorganic analysis, Replicate Error Ratio for radiochemistry analysis, Col. 31). This field will be populated for result identifier types LCS, RCT, Sur, MS, MSD, and REP.

Col. 17 Result Identifier	**Col. 28** Percent Recovery	**Col. 29** QC Upper Limit	**Col. 30** QC Lower Limit	**Col. 31** Relative Percent Difference or RER
MS	112	125	75	
Sur	106	121	70	
MSD	105	25	0	6.45
REP (rad)		.99	0	.50
REP (non-rad)		20	0	5.00

31) **Relative Percent Difference or Replicate Error Ratio (8)** The bias between the MS and MSD, or SA and REP. This field will be populated for result identifier types MSD and REP.

32) **Unit of Measure (8)** The unit of measure for the result. For each requested analysis the units must be consistent between the sample data and the associated QC data. Inorganics should be reported in “mg/kg” for SOIL and “mg/l” for AQUEOUS, unless otherwise specified on the ARCOC. This field must be filled for every record except surrogates, and it must match Appendix D.

33) **Result Qualifier (5)** This field will only be used when appropriate and must match Appendix G.

34) **Radiochemistry Uncertainty (10)** Error data is provided for Radionuclide analyses only. Uncertainties will be reported in the same format and units as given for the result, with the exception that plus/minus signs will not be used. Scientific notation may be used for radionuclide analysis. This field must be filled for every RAD record.

35) **QC Lot Identifier (18)** The cross-reference of QC batch and sample grouping. This field must be filled for every record.

36) **Detection Limit (10)** The detection limit specified for the analysis type as required in the test method. For diluted samples, use detection limit corrected for dilution factor. MDA should be used to report RAD detection limits, and PQL should be used to report non-RAD detection limits. This field is required for all records except Sur.

37) **Detection Limit Type (3)** The type of detection limit specified for the analysis method. This field is required for all records except Sur, and it must match Appendix E. The detection limit unit of measure must match the result unit of measure.

38) **Method Detection Limit (MDL) (10)** The method detection limit at the time of analysis. This field is required for all records except surrogates. Use the Critical level Concentration Corrected (L_c) for RAD, MDL for organics and inorganics. The MDL or L_c unit of measure must match the result unit of measure. If the L_c or MDL is not available report the detection limits (Field 36). This field is required for all records except Sur**.**
39) **Counting Time (6) (Optional)** (Sec) The length of time the aliquot is measured. The count must be in seconds. For radiochemistry only.
40) **Aliquot Weight or Volume (4) (Optional)** The weight or volume of the aliquot used in the analysis. This field must be numeric**.** For radiochemistry only.

Attachment 11
SNL/SMO Electronic Deliverable Specification

Address List

Send Laboratory Diskettes to:

Sandia National Laboratories
Attn: Rita Kavanaugh, Dept. 4142, MS 0729
P.O. Box 5800
Albuquerque, NM 87185-0729
Phone (505) 284-2553
Fax (505) 844-3128

Contact person is subject to change. Updates to be provided as necessary.

Appendix A

Analysis Lab ID

CODE	DESCRIPTION
BWXT	BWXT Services, Inc.
CFA	Cape Fear Analytical
GEL	GEL Laboratories, Inc.
TACA	Test America California (Air)
TAD	Test America Denver
TASL	Test America St.Louis
TATX	Test America Austin Texas (Air)

Appendix B
Sample Matrix Types

CODE	DESCRIPTION
AIR	Samples of confined air (AIR)
AQUEOUS	All water samples (DE, DIW, FDIW, FGW, FW, GW, LEA, PW, SW, WGW, WW, W)
BIOTA	All animal tissues, bodies or composite samples, including mammals, insects, fish, reptiles and amphibians (B)
FILTER	Specifically applies to air filter samples (AF)
GAS	Gas samples (SG, GAS)
OIL	Oil Samples (OIL)
SLUDGE	Chemical sludge, mixtures of which are neither solid nor particularly liquid (SLUDGE)
SOIL	All soil and sediment (SOIL)
SOLID	Solid samples
VEGETATION	All plant life, in general (V)
WIPE	Any type of material used for wipe samples (WIPE)

Attachment 11
SNL/SMO Electronic Deliverable Specification

Appendix C
Result Identifier

CODE	**DESCRIPTION**
SA	Normal Sample not QC
BL	Field Blanks including trip blanks and equipment blanks
LCS	Lab Control Sample
MB	Method Blank
MSD*	Matrix Spike Duplicate
MS*	Matrix Spike
Sur	Surrogate
REP*	Sample replicate for inorganic and radiochemistry
RCT	Radiochemistry Chemical Tracer

* Do not include MS, MSD or REP data derived from samples not included in the associated SDG.

Attachment 11
SNL/SMO Electronic Deliverable Specification

Appendix D
Unit of Measure

CODE	**DESCRIPTION**
C/100ml	Colonies per 100 milliliters
mg/l	Milligrams per liter
uohms/cm	Microohms per centimeter
pCi/g	Picocuries per gram
pCi/kg	Picocuries per kilogram
pCi/l	Picocuries per liter
pCi/ml	Picocuries per milliliter
pCi/SA	Picocuries per sample
pCi/m3	Picocuries per cubic meter
pg/l	Picograms per liter
pH	pH units
ppb v/v	Parts per billion as a volume per volume ratio
ug/kg	Micrograms per kilogram
ug/l	Micrograms per liter
ug/WIPE	Micrograms per wipe, used for PCBs
%	For use with percent solids, percent moisture, etc. (not used for radiochemistry)
%REC	for use with percent recovery on Lab Control Samples, etc.
mg/kg	Milligrams per kilogram
mg/sample	Milligrams per sample

APPENDIX E
DETECTION LIMIT TYPE

CODE	**DESCRIPTION**
MDL	Method Detection Limit
PQL	Practical Quantitative Limit Required
MDA	Minimum Detectable Amount (RAD analysis)

APPENDIX F
PREPARATION TYPE

CODE	DESCRIPTION
DIS	Dissolved analysis on the filtrate only
SUS	Suspended analysis on the particulate only
TOT	Total analysis on the entire sample
LEA	Leached (Example: TCLP analysis)

APPENDIX G
QUALIFIERS

CODE	DESCRIPTION
B	Analyte found in the blank and the sample. Not for radiochemistry. Organics > MDL Inorganics > MDL or PQL
E	Concentration exceeds calibration range of instrument and/or estimated quantity due to matrix interference. For organic analysis only.
H	Analytical Holding Time was exceeded. For all analysis if applicable.
h	Sample extraction or prep holding time was exceeded. For all analysis if applicable.
I	Interference, dilution was performed, detection limits are elevated. Not for radiochemistry.
J	Estimated value < PQL but greater than MDL. Not for radiochemistry.
U	Undetected analyzed but not detected. For Organics: the result is less than the MDL. For Inorganics: the result is less than the MDL or PQL. For RAD: the result is less than the MDA.
N	Results associated with a spike analysis that was outside control limits. For all analysis if applicable.
*	Results associated with a replicate analysis that was outside control limits, includes MSD and REP. For all analysis if applicable.
X	The "X" qualifier is used only to denote the existence of presumptive evidence suggesting that the reported analyte is not present in the sample. That is, this qualifier may be used only to indicate that the chemist believes the result to be a false positive.

APPENDIX H
SAMPLE DISPOSITION

CODE	**DESCRIPTION**
RECD, LOST	Received by lab and then lost
RECD, NOT ANAL	Received by lab but not analyzed
EXCEEDS HOLD TM	Sample exceeded holding time
WRONG PRESERVE	Sampling team used the wrong preservative
ANALYSIS FAILED	Lab unable to obtain reportable results
LAB CHANGED	Sample was shipped to a different lab
CANCELED	Received by lab then canceled
NO RESULT	Lab could not obtain a result for this analyte
CHANGE REQUEST	Lab requested to perform analysis not on ARCOC
NOT REQUESTED	Lab changed analysis without written approval

Note: Report all dispositions per analytical test

SANDIA NATIONAL LABORATORIES
LONG TERM STEWARDSHIP DEPARTMENT (4142)

QUALITY ASSURANCE PROJECT PLAN (QAPP) for the SAMPLE MANAGEMENT OFFICE

SMO-QAPP
Revision 3

Prepared By: 11-05-2013
Lorraine Herrera — Date
SMO Project Coordinator, 4142

Prepared By: Kevin A Lambert 11.05.2013
Kevin Lambert — Date
Technical Support Specialist, 4142

Reviewed By: Bonni Little 11-6-2013
Bonnie Little — Date
SMO Quality Assurance Coordinator, 4142

Reviewed By: Linda Gonzales 11/7/13
Linda Gonzales — Date
Waste Certification Official, 4144

Approved By: Pam Puissant 11/12/13
Pam Puissant, — Date
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: 11/12/13

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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Table of Contents

LIST OF ATTACHMENTS

Attachment I	Sample Management Office Functional Chart
Attachment II	Sample Management Office Sample Coordination and Packaging Process
Attachment III	Sample Management Office Sample/Financial Data Tracking Process
Attachment IV	Sample Management Office Analytical Data Proces

Revision History

Revision	Effective Date	Summary of Changes
0	December 18, 2003	New document.
1	March 28, 2007	Compare previous revision for changes.
2	April 9, 2010	Compare previous revision for changes.
3	November 12, 2013	Updated language to reflect current program elements and requirements. Attachments were revised.

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ACRONYMS AND ABBREVIATIONS

AOP	Administrative Operating Procedure
APHA	American Public Health Association
ARCOC	Analytical Request Chain of Custody
ASTM	American Society for Testing and Materials
CFR	Code of Federal Regulations
CFRC	Custom Funded Records Center
CLP	Contract Laboratory Program
CVR	Contract Verification Review
DOE	U.S. Department of Energy
DOT	U. S. Department of Transportation
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EPA	U.S. Environmental Protection Agency
EDMS	Environmental Data Management System (STAR – Analytical Results)
ES&H	Environment, Safety and Health
IATA	International Air Transportation Administration
ISO	International Organization for Standardization
LOP	Laboratory Operating Procedure
LQAP	Laboratory Quality Assurance Plan
NNSA	National Nuclear Security Administration
OP	Operating Procedure
PE	Performance Evaluation
OSHA	Occupational Safety and Health Administration
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
RPPM	Radiological Protection Procedures Manual
SAP	Sampling and Analysis Plan
SNL	Sandia National Laboratories
SNL/NM	Sandia National Laboratories/New Mexico
SMO	Sample Management Office
SOW	Statement of Work
SPF	Sample Packaging Facility
STAR	Sample Tracking and Analytical Results

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1.0 INTRODUCTION

1.1 GENERAL INFORMATION

This document provides the quality assurance (QA) requirements for activities performed by the Sandia National Laboratories, New Mexico (SNL/NM) Sample Management Office (SMO). Part A contains program elements and requirements from U.S. Department of Energy (DOE) 414.1D, *Quality Assurance*, and the Code of Federal Regulations (CFR), 10 CFR 830 Subpart A, *Nuclear Management*, *Quality Assurance Requirements*. This meets Corporate Process CG100.5, *Ensure Quality*. This Quality Assurance Project Plan (QAPP) provides information that conforms to U.S. Environmental Protection Agency (EPA) QA/R-5, *EPA requirements for QA Project Plans*. Additional QA guidance is provided in other QA documents from SMO customer programs, (i.e., Environmental Restoration, Environmental Life-cycle Management, Waste Management, Environmental Monitoring, and facilities Decontamination and Demolition programs). These documents are upper-tier documents to this QAPP. Waste management documents include, radioactive, mixed, and hazardous waste protocols.

The mission of the SMO is to provide centralized management of samples and analyses performed by contract laboratories. The primary QA objective of the SMO is to ensure that data is of adequate technical quality and content to meet programmatic data quality objectives (DQO).

For the purpose of this QAPP the words “other programs” mean various Waste Management, Environmental Monitoring, Facilities Decontamination and Demolition, and other sampling programs at SNL/NM.

2.0 Part A: DOE/SNL PROGRAM REQUIREMENTS

2.1 CRITERION 1, PROGRAM

2.1.1 Background, Purpose, Scope and Ownership

The purpose of Part A of this document is to meet requirements of QA activities within SNL/NM. This QAPP provides pointers to other QAPPs or lower tier plans and procedures written to meet project specific requirements as identified by external regulators. Lower tier activities either fall within the scope of this QAPP, or are covered by an appropriate QA program.

The SMO coordinates with customers to have samples analyzed for potential contaminants. Data from high-quality analyses is essential for environmental regulatory decision-making and critical for data defensibility. Commercial laboratories under contract to SMO perform analysis of samples. The SMO is responsible for procuring the contracts and continuously monitoring the data deliverables (both hardcopy and electronic) for compliance with contract and customer requirements. In order to assess the technical performance of the contract laboratories, performance evaluation samples are submitted and annual audits are performed. The SMO conducts contract verification review, electronic data processing, and manages the data validation process in accordance with SNL procedures. Additionally, the SMO provides sample packaging management services, acts as the liaison between each program and the contract laboratories and provides sample information management and data base maintenance for sample tracking and analytical results. Other tasks performed by the SMO may include sampling planning and coordination and sampling services.

Although the SMO provides sample and data management, it is not the owner of the samples or data. The final disposition of samples and data is the responsibility of the other programs that generate the samples.

During the lifetime of this QAPP, it is anticipated that some of the requirements and procedures of the SMO may change. This QAPP will be updated every three years, and changes provided to appropriate customers as required for information, concurrence, or approval. The SMO owns this document. The SMO is responsible for preparing, revising and distributing this document as necessary.

2.1.2 Applicability

This document applies to all SMO activities as outlined in Section 2.1.1.

The requirements set forth in this QAPP are applicable to personnel involved in the SMO as SNL/NM employees or as contractors. Sub-contracted activities are to be done in accordance with contract requirements, this QAPP, or covered by an appropriate QA program.

2.1.3 Requirements

SNL applicable policies, procedures, regulatory requirements, and DOE guidance that must be considered when planning and implementing activities can be found in this QAPP or other program QAPPs.

2.1.4 Functional Structure

The functional structure for the SMO is presented in Attachment I and includes position titles and lines of authority at and below the SMO Technical Lead level. Position titles, lines of authority, and management processes including planning, scheduling, and providing resources for work can be found in SNL Facilities, Financial and Human Resources corporate processes and procedures: FAC100.1, FIN100.2, HR100.1, and HR100.2.

2.1.5 Functional Responsibilities and Levels of Authority

SMO personnel are responsible for adherence to requirements stipulated in this QAPP that are applicable to their specific task(s). Each individual has an obligation to identify and act towards resolving conditions adverse to quality.

SMO positions with additional programmatic responsibilities are identified below.

SMO Department Manager

The SMO Department Manager is responsible for providing programmatic guidance leading to the development of this QAPP and the following:

- Reviewing and approving the QAPP.
- Acting as liaison to DOE and the National Nuclear Security Administration (NNSA)/Sandia Field Office on sample management issues.
- Ensuring that resources are available to perform tasks in compliance with this QAPP.

SMO Technical Lead

The SMO Technical Lead is responsible for the operations and activities conducted within the SMO, including those concerned with the implementation of the applicable requirements of this QAPP. The principal responsibilities of the SMO Technical Lead include the following:

- Managing contractor laboratory services, including procurement, acting as the Sandia Delegated Representative, reviewing routine performance assessments, and conducting general laboratory oversight.
- Developing and maintaining the SNL/NM *Contract Statement of Work (SOW) for Analytical Laboratories* that meets other programs' requirements.
- Developing and maintaining this QAPP.

SMO QA Coordinator

The principal responsibilities of the SMO QA Coordinator include the following:

- Providing guidance and expertise in the areas of quality assurance/quality control (QA/QC) relating to operations of the SMO.

- Interfacing with the Records Management Coordinator for maintenance of project documentation, and resolving record management concerns for storage and maintenance of sampling and analysis records.
- Ensuring that sufficient quality checks are in place to maintain the integrity of the SMO sample information management and analytical result databases.
- Facilitating implementation of QA requirements for the SMO.
- Assisting in developing and reviewing SMO procedures and this QAPP.
- Reviewing non-conformances and initiating corrective actions.

Project Coordinator

The Project Coordinator is responsible for coordinating efforts associated with SMO analytical services. The principal responsibilities of the Project Coordinator include the following:

- Acting as a point of contact between Task/Project Leaders and the analytical laboratories.
- Obtaining appropriate sample containers from a vendor or analytical laboratory.
- Scheduling projects with contract laboratories.
- Notifying analytical laboratories of any QA, environmental, safety, health, and sample matrix requirements regarding sample handling, preparation, and analysis.
- Resolving problems, issues, non-conformances, and errors for projects with regard to analytical data.
- Performing contract verification review (CVR) to ensure appropriate quality control (QC) analyses have been performed in accordance with the SNL/NM *Contract SOW for Analytical Laboratories*.
- Performing QC of data entered into the SMO database
- Performing electronic data QC and transfer.
- Processing and follow-up on any data package corrections, both hardcopy and electronic.
- Providing technical guidance and information, as required.
- Reviewing, verifying, and processing proformas and invoices from contractors.

Database Administrator

The principal responsibilities of the Database Administrator include the following:

- Designing, operating, and maintaining the SMO database.
- Reviewing and implementing improvements to system performance, establishing system design, and determining the need for changes to the design.

- Performing system backup and restoration functions.
- Developing new forms and reports with the Data Administrator.
- Resolving error messages generated by Oracle™.
- Establishing user accounts, passwords, and privileges within Oracle™.
- Submitting requests to administrative and support staff to establish new user accounts on the LAN.
- Assisting the SMO Technical Lead in developing electronic data deliverable (EDD) specifications for incorporation into the SNL/NM *Contract SOW for Analytical Laboratories*.
- Interfacing with SNL corporate computing regarding computer operations.

Project/Data Management

The principal responsibilities of project/data management staff include the following:

- Providing financial management support to the SMO by partnering with the center financial analyst and Program/Project Leaders.
- Tracking project/task expenditures.
- Ensuring compliance with the *SMO Data Management Plan*, Administrative Operating Procedure (AOP) 95-44.
- Tracking and maintaining sample information and performing QC on Analytical Request Chain of Custody (ARCOC).
- Receiving and processing 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.
- Reconciling data coordination QA concerns with Task/Project Leaders SMO staff, analytical laboratories, etc.
- Performing data entry including processing corrections.
- Providing backup project status reports.
- Submitting requests to the SNL Customer Funded Records Center (CFRC).

Sample Coordination and Packaging

The principal responsibilities of the sample coordination and packaging staff include the following:

- Overseeing the day-to-day operations of the SMO Sample Packaging Facility (SPF).

- Maintaining the SPF.
- Logging in and packaging of samples, as required.
- Developing and maintaining sample packaging procedures and operations in accordance with the SNL Integrated Safety Management System (ISMS) and *Sample Handling, Packaging and Shipping,* Laboratory Operating Procedure (LOP) 94-03.
- Ensuring samples are properly stored and packaged for shipment to the analytical laboratories in accordance with this QAPP and DOE, U.S. Department of Transportation (DOT), and International Air Transportation Administration (IATA) regulations.
- Ensuring that sample custody is properly maintained and documented in accordance with this QAPP and related Operating Procedures (OPs).
- Interfacing with SNL/NM Shipping, Radiation Protection Operations, and other SNL/NM on-site organizations.
- Maintaining facilities for preparation and return of samples.

Contract Laboratories

The principal responsibilities of the contract laboratories, reflected in applicable contracts, include the following:

- Developing and maintaining QA programs and procedures that meet the SNL/NM *Contract SOW for Analytical Laboratories*.
- Performing analyses in accordance with the SNL/NM *Contract SOW for Analytical Laboratories*.
- Providing data in accordance with format requirements in the SNL/NM *Contract SOW for Analytical Laboratories*.

Laboratory Oversight/Data Validation Contractor

The principal responsibilities of the Laboratory Oversight/Data Validation Contractor, as reflected in the applicable contract, include the following:

- Performing laboratory oversight as directed by the SNL/NM SMO.
- Conducting visits to and technical system audits of contractor laboratories to ensure compliance with SNL/NM *Contract SOW for Analytical Laboratories*.
- Performing data validation in accordance with the applicable procedures.
- Communicating non-compliance issues to the SMO Technical Lead and/or SMO Project Coordinator(s).
- Verifying implementation of laboratory corrective action plans.

2.1.6 Supporting Documents

Supporting documents are listed in Section 4.0. These are normative documents or may demonstrate compliance with QA requirements.

2.2 CRITERION 2, PERSONNEL TRAINING AND QUALIFICATION

2.2.1 Requirements

SMO personnel shall be trained and qualified to ensure they are capable of performing their assigned work. Training and qualification must be specific to the types of tasks performed. Personnel shall be provided continued training as required to ensure that job proficiency is maintained. Training shall be updated to meet required frequency schedules when specified. Training requirements and qualification shall be documented. Requirements can be found in SNL Corporate Policy, the SNL corporate Training Education and Development System (TEDS) database, or activity-specific operating procedures .

Training may consist of formal classroom training, on-line web training, or on-the-job training.

2.2.2 Responsibilities

SMO personnel are responsible for adherence to training requirements stipulated in this QAPP and in applicable sub-tier documents. SMO personnel are required to read all applicable documents and procedures and sign the Authorized Users List as evidence.

SMO management is responsible for committing resources to facilitate the qualification and training processes, for defining qualification and training requirements for personnel applicable to their specific task(s), and for ensuring personnel meet appropriate requirements.

2.2.3 Personnel Qualification or Certification

Specific qualifications or certifications identified in requirement sources such as, but not limited to, DOE, DOT, IATA orders and regulations, shall be included in the training program. An assessment of the education, experience, and any special physical capabilities that are required for a job shall be completed and the requirements shall be included in personnel criteria for job selection.

2.2.4 Supporting Documents

Supporting documents are listed in Attachment V. These are normative documents or may demonstrate compliance with QA requirements.

2.3 CRITERION 3, QUALITY IMPROVEMENT

2.3.1 Requirements

Quality improvement requirements are included in all SMO processes. Requirements include establishing and implementing the following:

- Continuous customer feedback and open communication between SMO personnel, customers, and contract laboratories (refer to Attachments II, III, and IV for communication loops for each SMO process).
- Processes to detect and prevent quality problems.
- Methods that identify, control, and correct data, services, and processes that do not meet established requirements.

2.3.2 Compliance with Requirements

Assessments, quality checks, and other performance measures provide the mechanisms to demonstrate that items, services or processes meet their requirements within the SMO.

SMO is responsible for:

- Assisting in interpretation and clarification of appropriate regulations, orders, policies, and standards.
- Ensuring that QA assessments of the data validation contractor and laboratories are conducted on a systematic basis.
- Providing guidance on project-specific QA matters (i.e., acceptance criteria and verification of program efficiency and implementation) to ensure quality requirements are met.
- Ensuring that QA deficiencies are properly recognized and resolved.

2.3.2.1 Detection and Prevention Process

Detection and prevention processes used for quality improvement shall include:

- Periodic review and update of the SNL/NM *Contract SOW for Analytical Laboratories* and other SMO documents.

- Feedback from staff, internal and external customers to identify potential problems and to initiate corrective actions.
- Frequent project meetings to review QA information and data analyzed to identify areas needing improvement.
- Management assessments as discussed in Section 2.9.
- Internal assessments as discussed in Section 3.4.

2.3.2.2 Identification, Control, and Correction of Problems

Several types of documents and procedures shall be used for identifying, controlling, and correcting problems for quality improvement. These documents include:

- Corporate Corrective Action Procedures CG100.6.1 and CG100.6.6
- DOE NNSA *Model SOW for Analytical Laboratories*
- SNL/NM *Contract SOW for Analytical Laboratories*
- Contract Laboratory QA Plans (LQAPs)
- *Procedure for Completing the Contract Verification Review (CVR)*, SMO-05-03
- *Data Validation Procedure for Chemical and Radiochemical Data*, AOP 00-03
- Other AOPs, Sampling and Analysis Plans (SAP), and Health and Safety Plans (HASP).

2.3.3 Review and Analysis of Information to Generate Improvements

Quality problems and other quality–related information, both positive and negative, from various internal and external sources, should be reviewed and analyzed by the SMO QA Coordinator to identify improvement opportunities in the quality management system, processes, items, products or services. Implemented improvements will be reviewed annually during the management assessment (see Section 2.9) to ensure their continuing suitability and effectiveness.

2.3.4 Supporting Documents

Supporting documents are listed in Attachment V. These are normative documents or may demonstrate compliance with QA requirements.

2.4 CRITERION 4, DOCUMENTS AND RECORDS

2.4.1 Requirements

The SMO shall maintain records to document QA/QC activities and to provide support for possible evidential proceedings. Records that provide documentary evidence of quality shall be specified, prepared and maintained in accordance with SNL/NM record-keeping procedures.

SMO records shall be transferred to the customer as well as to the Records Center for cataloging and storage in accordance with SNL Corporate Policy, IM100, *Information Management & Cyber Security*, DOE requirements, and the document control requirements of International Organization for Standardization (ISO) 9001 and ISO 14001.

2.4.2 Compliance with Requirements

Documents shall be reviewed to assure conformance with general corporate or organizational policies. The configuration of documents should be managed in accordance with AOP 09-11, *ES&H/Emergency Management Center Document Control Procedure*. The SNL/NM *Contract SOW for Analytical Laboratories* should be reviewed and updated as needed. This QAPP and applicable data review procedures shall be reviewed and updated to reflect significant changes every three years or as needed. Other relevant documents should be reviewed and updated as needed.

2.4.3 Documents and Records for Interfacing Organizations

Documents and records of interfacing organizations may include those imposed on the SMO, such as those described in the *Radiological Protection Procedures Manual* (RPPM) (MN471016) or those specified in the SNL/NM *Contract SOW for Analytical Laboratories*. The SMO will verify compliance of record-keeping requirements.

2.4.4 Supporting Documents

Supporting documents are listed in Attachment V. These are normative documents or may demonstrate compliance with QA requirements.

2.5 CRITERION 5, WORK PROCESSES

2.5.1 Requirements

SMO management and customers shall agree upon SMO work scope and processes. SMO processes are well-defined for those activities whose failure can lead to undesirable consequences and are shown in Attachments II, III, and IV. Work is documented in lower tier documents and procedures (Attachment V). SMO processes do not include items that would require Suspect/Counterfeit control requirements. Also, SMO processes do not require QA review of Safety Software. Additional information can be found in Section 3.0 Part B of this QAPP.

2.5.2 Compliance with Requirements

OPs and desk instructions shall be used for routine work. Controlled copies of OPs and desk instructions shall be readily available to all personnel. Work activities shall be documented and records maintained. Administrative controls shall be implemented as a part of these processes to ensure that the likelihood of failure of the activities is appropriately small. There must be assurance of accuracy of the instruments or equipment used for work activities monitoring or related to data collection and reporting. Additional information can be found in Section 3.0 Part B of this QAPP.

2.5.3 Work Processes for Interfacing Organizations

The work process procedures performed by interfacing organizations are governed by the following documents:

- RPPM
- Environment, Safety and Health (ES&H) Procedures
- Procurement documents

2.5.4 Supporting Documents

Supporting documents are listed in Attachment V. These are normative documents or may demonstrate compliance with QA requirements.

2.6 CRITERION 6, DESIGN

2.6.1 Requirements

SMO processes do not include design tasks and are not subjected to these requirements.

2.6.2 Compliance with Requirements

SMO processes do not include design tasks and are not subjected to these requirements.

2.6.3 Design for Interfacing Organizations

Formal design activities are typically done as contracted activities. Design work, including changes, must incorporate applicable requirements and design bases.

2.6.4 Supporting Documents

SMO processes do not include design tasks and are not subjected to these requirements.

2.7 CRITERION 7, PROCUREMENT

2.7.1 Requirements

The SMO follows SNL corporate QA policies and procedures provided by the Sandia Purchasing Organization. Procurement documents shall include QA requirements. SMO laboratory and data validation contracts are designated "Quality Significant" according to SCM100.2.11, *Acquire Quality Significant Items*. Contracts for major or stand-alone activities may either include a program specific to the contracted activity or may rely on the contractor's own QA program. If separate QA programs are used, they must be reviewed and approved by the SMO and must include the appropriate grading for the activity. If the contracting documents do not specify a QA program to be followed, the SMO program is required.

2.7.2 Compliance with Requirements

The SNL Procurement Center follows applicable Procurement Instructions and relevant SNL Corporate policies and procedures for procuring property, materials, and services. These policies cover the following areas:

- Procurement documents,
- Supplier qualification,
- Supplier monitoring,
- Nonconformance and corrective action,
- Inspection, and
- Product documentation.

SMO procurements also adhere to these processes and the required grading requirements.

The SMO, in conjunction with SNL/NM Purchasing, manages analytical and data validation services contracts in support of other programs. SMO develops the SOW for analytical services based on customer requirements, generates requests for proposals, and develops scoring criteria with SNL/NM Purchasing. The SMO reviews and scores laboratory qualifications and coordinates pre-award assessments, as required. The SMO makes laboratory selection based on best value for both technical and price criteria with concurrence of SNL/NM Purchasing. After contracts are placed, SMO monitors laboratory performance through periodic assessments, performance evaluations, and ongoing data review. The SMO is responsible for cost accounting and financial management for all contracted services provided to the other programs through the SMO.

The SNL/NM *Contract SOW for Analytical Laboratories* contains details and requirements for analytical services provided to SNL/NM by contract laboratories.

2.7.3 Supporting Documents

Supporting documents are listed in Attachment V. These are normative documents or may demonstrate compliance with quality assurance requirements.

2.8 CRITERION 8, INSPECTION AND ACCEPTANCE TESTING

2.8.1 Requirements

Inspection and testing of specified items, services and processes shall be conducted using established acceptance and performance criteria aligned with contractual and customer requirements. Qualified personnel (section 2.2.3) shall conduct inspections and tests.

Acceptance requirements for analytical data are discussed in Part B, Section 3.5, Data Validation and Usability.

2.8.2 Compliance with Requirements

The SMO shall receive sampling kits (sample bottles and coolers) from the contract laboratories per the requirements described in the SNL/NM *Contract SOW for Analytical Laboratories*. The sampling kits shall be requested using a bottle order (refer to Attachment II). Sampling kits are inspected on receipt by the packaging support staff using the criteria described on the original bottle order. Containers and analytical data shall be inspected as outlined in Part B, Section 3.3.8 and Section 3.5.

All equipment used by the SMO shall be properly maintained and inspected prior to use. Equipment requiring calibration will be performed in accordance with the manufacturer's instructions against known standards that are traceable to a national standard such as National Institute of Standard and Technology (NIST), if available. Malfunctioning equipment shall be clearly identified as being out of service and shall not be returned to service until it is demonstrated that the equipment is functioning properly.

The SMO will monitor contract laboratory services in accordance with the SNL/NM *Contract SOW for Analytical Laboratories*.

2.8.3 Inspection and Acceptance Testing by or for Interfacing Organizations

Laboratory instrument and equipment testing, inspection, and maintenance are addressed in Section 3.3. The SMO shall verify the adequacy of contractor inspection and testing procedures during the assessment process.

2.8.4 Supporting Documents

Supporting documents are listed in Attachment V. These are normative documents or may demonstrate compliance with QA requirements.

2.9 CRITERION 9, MANAGEMENT ASSESSMENT

2.9.1 Requirements

SMO staff, as well as management personnel from programs using SMO services, shall assess SMO processes. Problems that hinder the SMO from achieving its objectives shall be identified and documented. Management assessment shall include appropriate conclusions and suggest corrective actions. Management assessment shall be conducted in accordance with CG100.6, *Assure Performance Process* and CG100.6.19, *Conduct Management Review***.**

2.9.2 Compliance with Requirements

SMO staff and appropriate management personnel are responsible for management assessments and the documentation of results. The SMO shall provide assessment information to management personnel from programs using SMO services with appropriate conclusions and corrective actions as needed in compliance with CG100.6, *Assure Performance Process*.

2.9.3 Supporting Documents

Supporting documents are listed in Attachment V. These are normative documents or may demonstrate compliance with QA requirements.

2.10 CRITERION 10, INDEPENDENT ASSESSMENT

2.10.1 Requirements

The SMO shall promote the independent assessment process by assigning appropriate personnel to assist in any independent assessment and shall track and correct any non-conformances identified during this process in compliance with CG100.6, *Assure Performance Process* and CG100.6.3, *Determine, Plan and Perform Assessments.*

2.10.2 Compliance with Requirements

The determination of SMO processes to be examined is to be made by the requesting management. Independent assessments of processes shall be performed in accordance with a process defined by the applicable management program in compliance with CG100.6, *Assure Performance Process*. Independent assessments shall be planned and conducted to measure item and service quality, to measure the adequacy of work performance and to promote improvement. The group performing independent assessments shall have sufficient authority and freedom from the line to carry out its responsibilities. The group shall also have access to the appropriate SMO management and staff.

2.10.3 Assessments Performed by External Organizations

Independent assessments may be performed periodically by a variety of independent groups outside of SMO. These assessments may be a part of other program's QA to assess operations and work processes.

2.10.4 Supporting Documents

Supporting documents are listed in Attachment V. These are normative documents or may demonstrate compliance with QA requirements.

3.0 Part B: DATA REQUIREMENTS

3.1 DATA ACQUISITION

The SMO has the primary responsibility of ensuring that the quality of the data generated for sampling programs meet data quality requirements necessary to determine and demonstrate compliance with DOE requirements and local, state and federal environmental regulations. This QAPP addresses applicable elements from EPA QA/R-5 as they apply to the SMO and contract laboratories.

3.2 PROJECT MANAGEMENT

Project management and applicable information from EPA QA/R-5 is addressed in Section 2.1 of this QAPP.

3.3 DATA GENERATION AND ACQUISITION

3.3.1 Sampling and Process Design

The DQO process provides a means of defining the appropriate quality of the data required to support environmental projects. Other programs define DQOs and ensure that they are met. The SMO is responsible for incorporating necessary and applicable requirements into the SNL/NM *Contract SOW for Analytical Laboratories* so that the other programs DQOs are met.

3.3.2 Sampling Method Requirements

After DQOs are developed for a particular activity, appropriate sampling strategies must be established. The SMO provides guidance to customers on sampling strategies.

Further discussion of sampling method requirements is presented in customer field operating procedures and activity-specific SAPs.

3.3.3 Sample Handling and Custody Requirements

The SMO shall have procedures that address the sample handling and custody requirements that apply to SMO-specific tasks or activities. These procedures will be readily available to all SMO personnel handling samples. The SMO shall ensure staff is trained in and follow the sample handling and custody requirements.

Each analytical laboratory participating in the analysis of SNL/NM SMO customer samples shall have Laboratory Operating Procedures (LOPs) that address activities related to sample custody (such as receiving, storing, and disposing of samples and maintaining the chain-of-custody records). The LOPs will be readily available to all appropriate laboratory personnel.

The analytical laboratories are solely responsible for lawful disposal of samples after the sample storage requirement is fulfilled. Detailed sample handling and custody requirements are discussed in the current revisions of the *Sample Management and Custody* procedure, AOP 95-16, SNL/NM *Contract SOW for Analytical Laboratories*, and DOE NNSA *Model SOW for Analytical Laboratories*.

3.3.4 Analytical Methods

Analytical, extraction, and other preparation methods are selected and specified in accordance with the DQOs and are identified in the SNL/NM Contract SOW for Analytical Laboratories. A general requirement is that industry-standard methods, such as EPA SW-846 (Third Edition), EPA 600 series methods, Occupational Safety and Health Administration (OSHA) methods, American Society for Testing and Materials (ASTM) methods, and standard methods be used where possible. Applicable standard methods are used when appropriate or SNL/NM approved, laboratory specific methods may be substituted. New methods may be developed when required. All analytical methods must be pre-approved by the SNL/NM SMO, documented in a laboratory-approved LOP, and performed as specified in a controlled laboratory setting. Analytical results (organic, inorganic, radiochemical, and miscellaneous analytes) and non-analytical data shall be reported in the units consistent with the specified method.

The SMO shall verify that the analytical laboratories follow the SNL/NM *Contract SOW for Analytical Laboratories* as it pertains to Standard Methods. The standard methods requirements are discussed in the SNL/NM *Contract SOW for Analytical Laboratories*, DOE NNSA *Model SOW for Analytical Laboratories*, EPA SW-846 and other EPA documents, OSHA, ASTM, and American Public Health Association (APHA) documents.

All analytical laboratories will maintain controlled copies of approved LOPs for each analytical method or general procedure in accordance with the DOE NNSA *Model SOW for Analytical Laboratories*.

3.3.5 Quality Control

Laboratory analytical activities shall be subjected to QC checks using QC samples. QC sample requirements are specified in the SNL/NM *Contract SOW for Analytical Laboratories*, LQAPs, LOPs and in the published test method.

3.3.6 Instrument/Equipment Testing, Inspection, and Maintenance

Each analytical laboratory shall have procedures that address activities related to instrument/equipment testing, inspection and maintenance. Laboratory equipment shall be inspected, tested, and maintained in accordance with LOPs, LQAPs, and the SNL/NM *Contract SOW for Analytical Laboratories*.

Regular inspection and calibration is performed to ensure that the fume hood used for sample container preparation is operating correctly according to manufacture specifications.

Maintenance is performed annually per the corporate schedule for inspection and calibration site wide. See references in procedure SMO-SH-A-001, Fume Hood Usage and Sample Container Preparation, current revision.

3.3.6.1 Preventive Maintenance Documentation

Analytical laboratory preventive maintenance activities shall be documented, and the records maintained in accordance with LOPs, LQAPs, and the SNL/NM *Contract SOW for Analytical Laboratories*.

3.3.7 Instrument/Equipment Calibration and Frequency

Each laboratory shall have procedures that address instrument/equipment calibration and frequency requirements. Instruments shall be calibrated in accordance with the SNL/NM *Contract SOW for Analytical Laboratories,* LOPs, LQAPs, and applicable analytical test method.

3.3.8 Inspection/Acceptance of Supplies and Consumables

The SMO shall ensure staff is trained in sampling kit (sample containers and cooler) requirements. SMO shall inspect sampling kits to determine that they are undamaged, match the project-specific bottle order and meet any other specific written requirements associated with the SNL/NM *Contract SOW for Analytical Laboratories*. Any errors or damage to shipping containers will be addressed in accordance with the SNL/NM *Contract SOW for Analytical Laboratories* and procurement policies.

3.3.9 Non-direct Measurement

Data and information gathered from sources outside the SMO or gathered from any source that was not collected under an approved QA program appropriate to that used for SMO shall not be approved for SMO use. Indirect measurement data reported by contract laboratories, as a result of data produced through direct measurements, shall be reviewed and designated usable if all other QC associated with the direct measurement data are acceptable.

3.3.10 Data Management

The SMO tracks sample data generated from field, shipping, analysis, data review, and validation activities. SMO data management functions include tracking sample shipments to the analytical laboratories, tracking supporting and analytical data returned from the analytical laboratories and cost accounting associated with sample analysis and data validation. The SMO receives and tracks both hardcopy data and EDD files from the analytical laboratories. The EDD file is checked for accuracy and compared to hardcopy data in accordance with SMO *Procedure for Electronic Data Deliverable (EDD) Processing,* SMO-05-04 and the SNL/NM *Contract SOW for Analytical Laboratories*. When data is manually entered, the hardcopy data is used and verified.

The SMO shall verify that the laboratories follow the SNL/NM *Contract SOW for Analytical Laboratories* as it pertains to data management. The analytical laboratory certifies the laboratory-generated data in accordance with the SNL/NM *Contract SOW for Analytical Laboratories*, EPA SW-846, and other standard methods. A summary report that includes analytical and QC results is prepared and approved by the analytical laboratory. Data packages shall conform to contract or procedural requirements. Data should also be validated by independent review as described in Part B, Section 3.5.

Data shall be managed through the Environmental Data Management System (EDMS) and the Sample Tracking and Analytical Results (STAR) database in accordance with the current revision of the *SMO Data Management Plan,* AOP 95-44, SMO *Procedure for Completing the Contract Verification Review (CVR),* SMO-05-03, SMO *Procedure for Electronic Data Deliverable (EDD) Processing,* SMO-05-04, and in the SNL/NM SMO *Data Validation Procedure for Chemical and Radiochemical Data,* AOP 00-03.

3.4 ASSESSMENT AND OVERSIGHT

Management and quality assurance personnel shall complete assessments of QA related activities done at various levels in the SMO organization. Assessments include but are not limited to surveillance, data audit or assessment, system audits, limited-scope audits, management review, or readiness review.

Management assessments are addressed in Part A, Section 2.9 of this QAPP. For additional details, see CG100.6, *Assure Performance Process,* and Criterion 3, Section 2.3, requirements in this QAPP.

3.4.1 Assessment and Response Actions

The SMO assessment/surveillance teams shall include personnel with the necessary expertise and knowledge of SMO processes and laboratory operations to address the requirements established in this QAPP, the SNL/NM Contract SOW for Analytical Laboratories, and other relevant documents. The SMO Technical Lead, or designee, functions as the team leader and shall be responsible for the selection of assessment/surveillance team members. Assessors shall be independent of any direct responsibility for performance of the activities that they assess. The assessors shall have the authority to stop work based on quality or safety issues

Assessments will be regularly executed as part of SMO routine operations. The SMO Technical Lead should participate in system and performance assessments of each contract laboratory at least once during the contract term. Surveillances may be conducted at any time as determined by project requirements or in response to conditions perceived as potentially adverse to quality.

Assessment records shall include worksheets, reports, corrective action requests (if necessary), written replies, and a record of completion of corrective actions.

3.4.1.1 Laboratory Performance Assessment

Laboratory performance assessments determine the accuracy of laboratory measurement systems and include annual laboratory audits, routine performance evaluation (blind spike analysis), and data package assessments. Performance assessments shall be conducted as off-site data are generated, reduced, and analyzed. All laboratories providing support to the SNL/NM SMO shall be subject to performance assessment requirements as specified in the SNL/NM *Contract SOW for Analytical Laboratories*. Preliminary assessments should be performed as needed.
In addition, the laboratory will conduct internal assessments to verify that its operations continue to comply with the requirements of the laboratory's quality systems. All quality systems will be audited at a minimum frequency of once per year in accordance with the LQAP.

Laboratories will be subject to one data package assessment annually in accordance with the DOE NNSA *Model SOW for Analytical Laboratories*. This may be conducted during the annual systems assessment or performed as a separate assessment. Additional data package assessments may be performed as deemed necessary by the SMO.

Laboratories shall participate in analysis of performance evaluation (PE) samples or assessment samples as required by the SMO, DOE, EPA, and/or the State of New Mexico performance evaluation programs (if required in the future). In addition, laboratories may be subject to the submission of PE samples from the SMO at any time in response to a corrective action, to evaluate the performance of a new method, or other non-routine situations. Analysis results should be compared to predetermined or calculated acceptance limits. Records of performance evaluation samples shall be maintained and any problems shall be identified, corrective actions taken and performance re-evaluated prior to analysis of additional applicable samples.

3.4.1.2 Laboratory System Assessment

System assessments verify the application of the QA system and evaluate the level of compliance with the system. System assessments for SMO cover laboratory activities and final reports. Work areas, activities, activity documentation, and QA/QC procedures and the effectiveness of their implementation shall be evaluated. All analytical laboratories providing support to the

SNL/NM SMO shall be subject to system assessment requirements as specified in the SNL/NM *Contract SOW for Analytical Laboratories*.

Prior to implementation of new off-site analytical services contracts, a pre-award assessment of the laboratory(s) shall be performed. Laboratory system assessments during the course of a contract shall include an on-site visit to the analytical laboratory(s) by SNL/NM representatives or designees.

Technical assessments to verify adherence to requirements as stated in the SNL/NM *Contract SOW for Analytical Laboratories* shall be conducted on all laboratories generating data used for regulatory compliance and decision-making purposes.

3.4.1.3 Assessment Documentation

Assessment results shall be formally documented by personnel and reported by the SMO Task Leader in accordance with the SNL/NM *Contract SOW for Analytical Laboratories* and relevant project requirements. In the event that the lead assessor is not the SMO Technical Lead, the SMO Technical Lead shall review and approve the assessment report. An assessment report contains any observations, findings, and associated corrective actions. Assessment reports shall be maintained as part of the program files and archived in the SNL CFRC when the contract has ended. Assessment and surveillance reports are considered public documents.

3.4.1.4 Response Actions

The results of assessments shall be entered into a corrective action system in compliance with CG100.6, *Assure Performance Process*.

In addition, personnel shall be responsible for identifying and reporting deficiencies and initiating the corrective action process. Documentation of nonconforming items or processes should typically be on a nonconformance record or other forms intended to detail the circumstances of the deviation.

The responsibility for monitoring the quality of analytical systems lies with contract analytical laboratory personnel. All corrective activities resulting from deficiencies occurring at the analytical laboratory shall comply with the LQAP and the SNL/NM *Contract SOW for Analytical Laboratories*. Additionally, the analytical laboratory shall notify the SMO of the deficiency and, if possible, identify potential causes and corrective action.

Deficiencies shall be reported and corrective action initiated by the SMO or contracted analytical laboratories if any of the following conditions arise:

- Specific requirements of the analysis method or LOPs are not met,
- Data quality measurements for precision, accuracy and completeness are not achieved, or
- Lab data review indicates that data are incomplete, that improper calculations were performed, incorrect methodology or technique was employed, or that an instrument malfunction has occurred.

When corrective actions are required, it shall be the responsibility of the analytical laboratory to provide a Corrective Action Report that details planned action to correct the findings and a schedule for completion. The SMO Technical Lead, or designee, shall document that assessment findings are resolved and that the appropriate corrective actions have been implemented in a timely manner. The SMO Technical Lead shall attempt to resolve any disagreements or disputes related to assessment or surveillance findings. Root cause analysis, in compliance with CG100.6, *Assure Performance Process,* shall be completed to identify the actions necessary to prevent recurrence of the condition whenever the SMO Technical Lead decides that the severity or recurrence of a deficiency indicates it is needed. If a satisfactory resolution cannot be reached, the issue shall be elevated to the next level of contract analytical laboratory management.

See Part A, Sections 2.9 and 2.10 of this QAPP and applicable AOPs for further details on the methods by which deficiencies are identified and corrected.

3.4.2 Reports to Management

Management shall be kept apprised of project status and events impacting quality, both informally and formally. Open channels of communication shall be fostered among SMO staff, customers, and management at all times. Regularly scheduled status reports shall include a discussion of quality activities, if any. Such activities may include periodic assessment of the precision, accuracy, and completeness of measurement data, the results of any surveillance or audits and any QA problems.

The SMO shall provide reports of results of any QA/QC activities and documentation associated with the handling, shipping and analysis of samples to the Task/Project Leaders and Department Managers.

The SMO Technical Lead shall ensure that the Department Managers and customers are kept informed of, and have access to, results of system and performance assessments and data-package review activities. Any programmatic QA issues identified that adversely affect the quality of data generated shall be reported to the Department Managers immediately.

3.5 DATA VALIDATION AND USABILITY

This section discusses guidelines for assessing data quality. QA protocols are presented for data reduction, verification, validation, and reporting activities performed as part of the SMO function.

See applicable AOPs and activity-specific QAPPs for further details on data validation and usability.

3.5.1 Data Review, Verification, and Validation

Procedures for assessing data quality, data reduction, verification, validation, and reporting activities shall comply with applicable requirements described as part of EPA SW-846 and other Standard Methods, LQAP, LOPs, the SNL/NM *Contract SOW for Analytical Laboratories*, the DOE NNSA *Model SOW for Analytical Laboratories*, and AOPs.

3.5.1.1 Laboratory Data Review

The analytical laboratory shall complete quality reviews of all data packages as specified in the SNL/NM *Contract SOW for Analytical Laboratories* prior to submitting them to the SMO.

The SMO shall verify that the contract analytical laboratories follow the SNL/NM *Contract SOW for Analytical Laboratories* relative to laboratory data review. See EPA SW-846 and other Standard Methods, LQAP, LOPs, the SNL/NM *Contract SOW for Analytical Laboratories*, the DOE NNSA *Model SOW for Analytical Laboratories* and AOPs for further details on laboratory data review and reduction requirements.

3.5.1.2 Laboratory Data Verification and Validation

The specific criteria to be reviewed in the analytical laboratory data verification and validation process depend on the sample matrix, analytical method and applicable regulatory requirements. The initial responsibility for monitoring the quality of analytical data lies with the analytical laboratory analyst. The analyst shall verify that all QC procedures specified for each analytical method are followed and that the results of QC check sample analyses are within the acceptance criteria established for the method. When results are not within control limits, corrective actions shall be taken according to EPA SW-846 and other Standard Methods, the SNL/NM *Contract SOW for Analytical Laboratories*, the LQAP, LOPs, or SMO directive.

The SMO shall verify that the analytical laboratories follow the SNL/NM *Contract SOW for Analytical Laboratories* relative to laboratory data verification and validation. See EPA SW-846 and other Standard Methods, LQAP, LOPs, the SNL/NM *Contract SOW for Analytical Laboratories* and the DOE NNSA *Model SOW for Analytical Laboratories* for further details on the laboratory data verification and validation requirements.

SMO personnel are responsible for data verification and editing upon receipt of the data package. A CVR is conducted in accordance with SMO-05-03, *Procedure for Completing the Contract Verification Review (CVR).* The CVR checks completeness and compliance of the sample custody and laboratory report documentation, examines sample management and custody and checks technical, QC, and reporting requirements imposed upon the analytical laboratory through the SNL/NM *Contract SOW for Analytical Laboratories*. The CVR checklist provides the SMO with a record of analytical laboratory performance on each data package and allows for SMO tracking of reported deficiencies, correction requests and problem resolutions. The SMO, using the results from the CVR, monitors the performance of contracted analytical laboratories and in accordance with the SNL/NM *Contract SOW for Analytical Laboratories* adjusts payment.

The SMO shall perform a QC check on the EDD and process the file for loading into the analytical results database. This review is performed using SMO-05-04, *Procedure for Electronic Data Deliverable (EDD) Processing*. During the EDD processing, the file is compared to the hardcopy data to ensure accuracy and checked for compliance with the EDD Specification found in the SNL/NM *Contract SOW for Analytical Laboratories*. The SMO documents deficiencies, requests corrections and resolves EDD problems.

The SMO shall perform data validation if requested to do so by the customer, using AOP 00-03, *Data Validation Procedure for Chemical and Radiochemical Data*. This AOP provides instructions for the qualification (known as validation) of common laboratory analytical data. This procedure is used to determine the quality and usability of chemical (organic and inorganic) and/or radiochemical analytical data acquired in support of other programs. This procedure generally follows the guidelines and approach presented in the EPA *Contract Laboratory Program (CLP) Functional Guidelines*, and in EPA SW-846 and other standard methods, with modifications made to address analyses requested by the other programs. A data validation report shall be completed by the SMO that includes information regarding the overall quality of the data and the resulting data qualifiers. Data validation qualifiers are then imported into the analytical results database.

Qualification of data performed under the data validation procedure (AOP 00-03) does not preclude the qualification of data by the analytical laboratories due to unexpected analytical uncertainty, nor does the validation review replace any data usability review for specific project use. For details on site-specific verification/validation requirements, see activity-specific QAPPs and SAPs.

3.5.1.3 Data Reporting

Analytical laboratory-generated data shall be reported on EDD and in hard-copy data reports as requested. All analytical laboratory data report packages for each type of analysis shall contain a case narrative that summarizes the laboratory analysis for the given set of samples. Complete data packages include sample data, QC summaries, and additional supporting data needed to perform data validation. Laboratory reporting requirements and report format shall be in accordance with the SNL/NM *Contract SOW for Analytical Laboratories*.

The SMO shall verify that the analytical laboratories follow the SNL/NM *Contract SOW for Analytical Laboratories* as it pertains to data reporting. Details on tracking data and entering it into the appropriate databases are presented in the *SMO Data Management Plan*, AOP 95-44, and the SMO *Procedure for Electronic Data Deliverable (EDD) Processing*, SMO-05-04, and other programs, procedures and documents.

All laboratory analytical reports shall be archived by the SNL CFRC.

3.5.2 Reconciliation with User Requirements

Task/Project Leaders shall review and analyze analytical laboratory-generated data prior to use and inclusion in reports. If sample results are unusable, they cannot, by definition, be used in the decision making process. If sample results that are critical to the decision making process are unusable, a determination must be made as to whether or not re-analysis or re-sampling is possible. Sample results that have restricted usability must be used cautiously in the decision making process with their restrictions clearly defined. The SMO shall work closely with analytical contract laboratories and customers to assure that laboratory-generated data meets DQOs and other data needs and that they are sufficient to support any decisions made.

4.0 SUPPORTING DOCUMENTS AND REFERENCES

AOP 95-16, *Sample Management and Custody*, current revision.
AOP 95-44, *SMO Data Management Plan*, current revision.
AOP 00-03, *Data Validation Procedure for Chemical and Radiochemical Data*, current revision.
AOP 09-11, *ES&H/Emergency Management Center Document Control Procedure*, current revision.
CG100.5, *Ensure Quality*.
CG100.6, *Assure Performance Process.*
CG100.6.1, *Manage Risks.*

CG100.6.3, *Determine, Plan and Perform Assessments.*
CG100.6.6, *Determine and Take Action.*
CG100.6.19, *Conduct Management Review.*
ESH100.2.RAD.1, *Implement Radiation Protection Procedures,* (MN471016) *Radiological Protection Procedures Manual,* current revision.
FAC 100.1, *Plan Real Property Assets.*
FIN 100.2, *Provide Financial Project Management Services.*
HR 100.1, *Acquire Talent.*
HR 100.2, *Develop the Workforce.*
IM100, *Information Management & Cyber Security.*
LOP 94-03, *Sample Handling, Packaging and Shipping*, current revision.
SCM100.2.11, *Acquire Quality Significant Items*.
SNL/NM, *Contract SOW for Analytical Laboratories,* current revision.
SMO-SH-A-001, *Fume Hood Usage and Sample Container Preparation*, current revision.
SMO-05-03, *Procedure for Completing the Contract Verification Review (CVR),* current revision.
SMO-05-04, *Procedure for Electronic Data Deliverable (EDD) Processing*, current revision.
DOE O 414.1D *Quality Assurance*, 2011.
DOE NNSA, *Model SOW for Analytical Laboratories*, current revision.
EPA QA/R-5, *EPA Requirements for Quality Assurance Project Plans*, 2001 (Reissued 2006).
10 CFR 830 Subpart A, *Nuclear Safety Management, Quality Assurance Requirements*.
ISO 9001, *Quality Management Systems – Requirements, 2008.*
ISO 14001, *Environmental management systems -- Requirements with guidance for use, 2004.*

Developmental References

The following have been used in the development of the Sample Management Office QA Program.

AOP 04-04, *ES&H and Emergency Management Self-Improvement Process*, current revision.
AOP 09-10, *Work Planning and Controls*, current revision.
CG100.6.7 *Conduct and Manage Audits*.
ESH100, *Environment Safety & Health Corporate Policy*.
ESH100.2.GEN.2, Determine, *Complete, and Document Required ES&H Training*.
ESH100.2.GEN.3, *Develop and Use Technical Work Documents*.
ESH100.4.RPT.3, *Report Occurrences*.
ESH100.4.FI.1, *Perform ES&H Line Self-Assessment Activities*.
ESH100.4.FI.3, *Implement and Manage Corrective Actions*.
HR100, *Human Resources Management Corporate Policy*.
HR100.3.8, *Manage and Evaluate Employee Performance*.
IM100.2.1, *Control of Documents*.
IM100.2.2, *Control of Records*.
IRN Web item, Summary of the Major Processes in the Financial Information System
SCM100.2, *Acquire Property, Material, and Services* (formerly the SNL Procurement Manual).
SNL, Financial Information System Database (FIS)
SNL, *Quality-Significant Procurement Handbook*, current revision.
SNL, *Records Management Manual, current revision*.
SNL, Site-wide Personnel Training Plan for Resource Conservation and Recovery Act – Regulated Waste Management Units, Appendix D, General Part B Permit Application, SNL/NM

DOE, DOECAP, *Quality Systems for Analytical Services (QSAS)*, current revision.
DOE G 414.1-2B, *Quality Assurance Program Guide*, 2011.
EPA, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846 (Third Edition), as updated. http://www.epa.gov/osw/hazard/testmethods/sw846/index.htm.
EPA 600 series methods.
EPA, *Methods for the Determination of Organic Compounds in Drinking Water*, EPA 600/4-88/039, 1988 (Revised 1991).
EPA, *CLP Functional Guidelines*, current revision.
EPA QA/G-4, *Guidance on Systematic Planning using the Data Quality Objectives Process*, 2006.
EPA QA/G-5, Guidance for *Quality Assurance Project Plans*, 2002.

APHA, *Standard Methods for the Examination of Water and Wastewater*, current revision.

ASTM, *Annual Book of ASTM Standards*, Volumes 3 and 11.
OSHA, *OSHA Analytical Methods Manual*, current revision.

Other Developmental References

DOE, see United States Department of Energy.

EPA, see United States Environmental Protection Agency.

Gilbert, R. O., 1987. *Statistical Methods for Environmental Pollution Monitoring*, Van Nostrand Reinhold Company, Inc., New York, NY.

SNL/NM, see Sandia National Laboratories/New Mexico.

Sandia National Laboratories/New Mexico (SNL/NM), *Environmental Management System Manual* (PG47022), current revision, Environmental Programs, Sandia National Laboratories, Albuquerque, New Mexico.

United States Department of Energy (DOE), 1997. *EML Procedures Manual*, (HASL-300), 28th edition (as updated). United States Department of Energy, Environmental Measurements Laboratory, New York, New York.

United States Environmental Protection Agency (EPA), 1992. *Hazardous Waste Permit for Sandia National Laboratory*, Permit Number NM5890110518 (as updated). United States Environmental Protection Agency, Washington, D.C.

EPA, 2009. *Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities*, Unified Guidance (EPA/530-R-09-007). United States Environmental Protection Agency, Office of Resource Conservation and Recovery, Washington, D.C.

EPA, 2007, *Guidance for Preparing Standard Operating Procedures (SOPs)*, EPA QA/G-6, EPA/600/B-07/001, United States Environmental Protection Agency, Office of Environmental Information, Washington, D.C.

EPA, 2005, *Waste Management System; Testing and Monitoring Activities; Final Rule: Methods Innovation Rule and SW-846 Final Update IIIB; Final Rule*, published in the Federal Register on June 14th, 2005 (70 FR 34538). United States Environmental Protection Agency, Office of Solid Waste, Washington, D.C.

EPA, 1997, *EPA Implementation Guide for the Code of Environmental Management Principles for Federal Agencies (CEMP)*, EPA/315/B-97/001, Enforcement and Compliance Assurance, Washington, D.C.
EPA, 1993. *Determination of Inorganic Anions in Water by Ion Chromatography, Method 300.0*, United States Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Inorganic Chemistry Branch, Cincinnati, Ohio.

EPA, 1989a. *Methods for Evaluating the Attainment of Cleanup Standards, Volume 1: Soils and Solid Media, EPA/230/02-89-042*, United States Environmental Protection Agency, Office of Policy, Planning, and Evaluation, Washington, D.C.

EPA, 1989b. *Soil Sampling Quality Assurance User's Guide, EPA/600/8-89-046*, 2nd ed., United States Environmental Protection Agency, Office of Research and Development, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

EPA, 1989c. *RCRA Facility Investigation (RFI) Guidance, Volume II of IV, Soil, Groundwater and Subsurface Gas Releases*, Interim Final, *EPA/530/SW-89-031*, Office of Solid Waste, Waste Management Division, Washington, D.C.

EPA, 1989d. *RCRA Facility Investigation (RFI) Guidance, Volume III of IV, Air and Surface Water Releases*, Interim Final, *EPA/530/SW-89-031*, Office of Solid Waste, Waste Management Division, Washington, D.C.

EPA, 1988. *Interim Report on Sampling Design Methodology, EPA/600/X-88-408*, United States Environmental Protection Agency, Environmental Monitoring Support Laboratory, Las Vegas, Nevada.

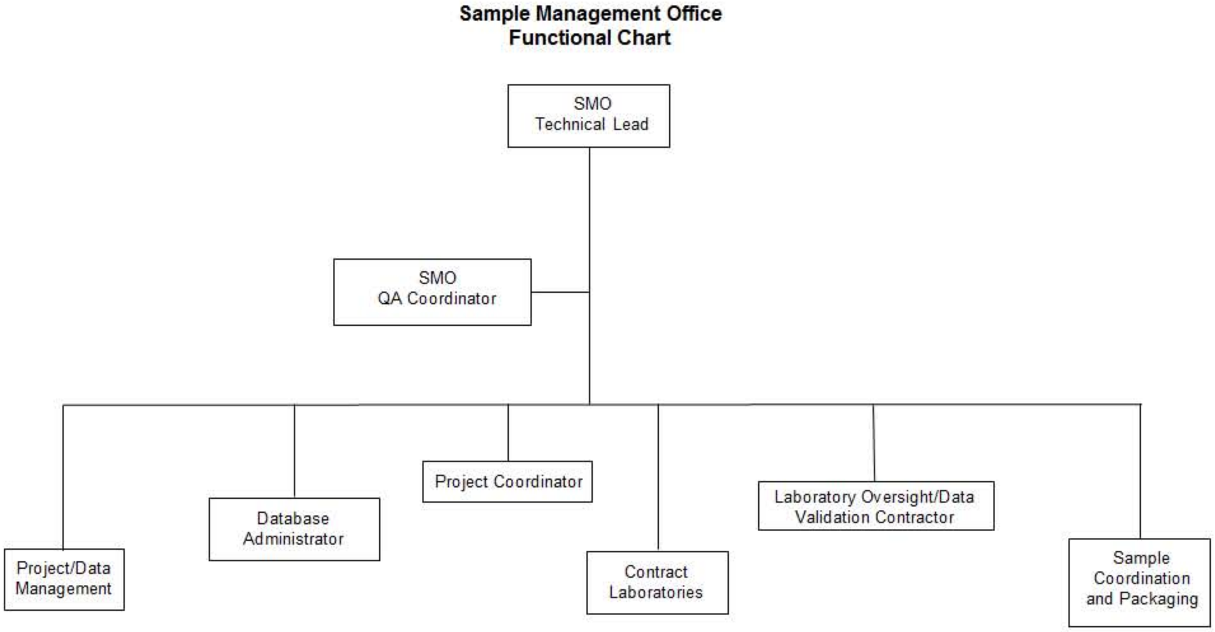
EPA, 1987a. *Data Quality Objectives Process for Superfund: Interim Final Guidance, EPA/540/R-93/071*, United States Environmental Protection Agency, Office of Emergency and Remedial Response and Office of Waste Programs Enforcement, Washington, D.C.

EPA, 1986d. *RCRA Groundwater Monitoring Technical Enforcement Guidance Document (TEGD)*, OSWER-9950.1, United States Environmental Protection Agency, Office of Waste and Emergency Response, Washington, D.C.

EPA, 1979. *Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA/600/4-79-019*, United States Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

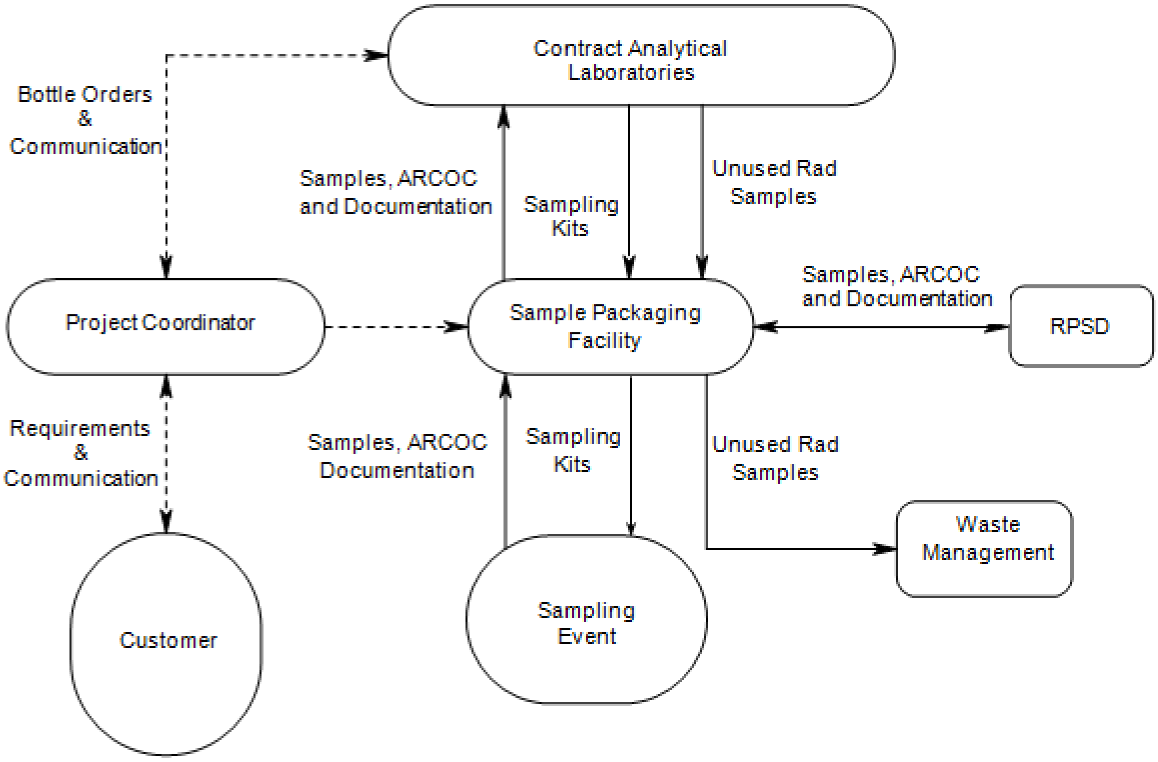
EPA, 1977. *Handbook for Analytical Quality Control in Radioanalytical Laboratories*, EPA/600/7-77-088, United States Environmental Protection Agency, Office of Research and Development, Office of Energy, Minerals and Industry, Washington, D.C.

Attachment I



Attachment II

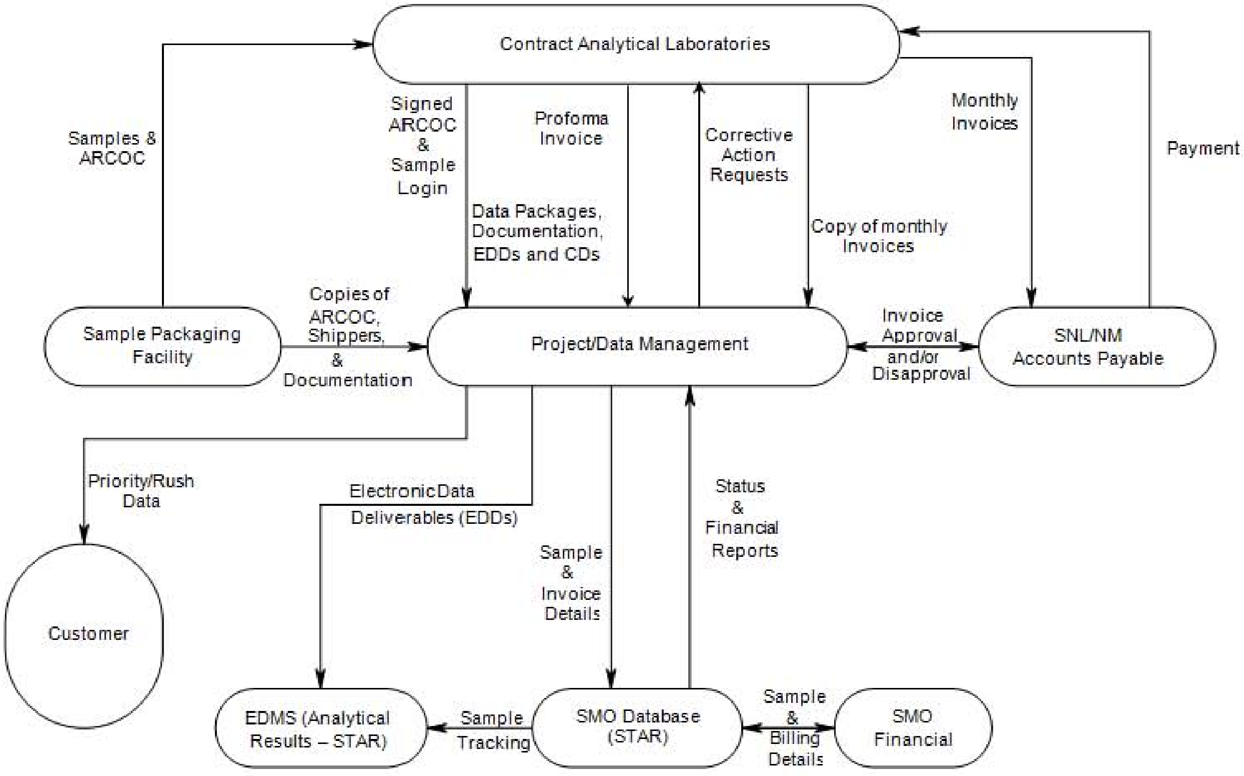
Sample Management Office
Sample Coordination and Packaging Process



*The unused rad samples that are returned are only those which the laboratories can’t dispose of, i.e. PCB mixed waste and Limited Quantity rad. The contract laboratories dispose of all other excess samples.

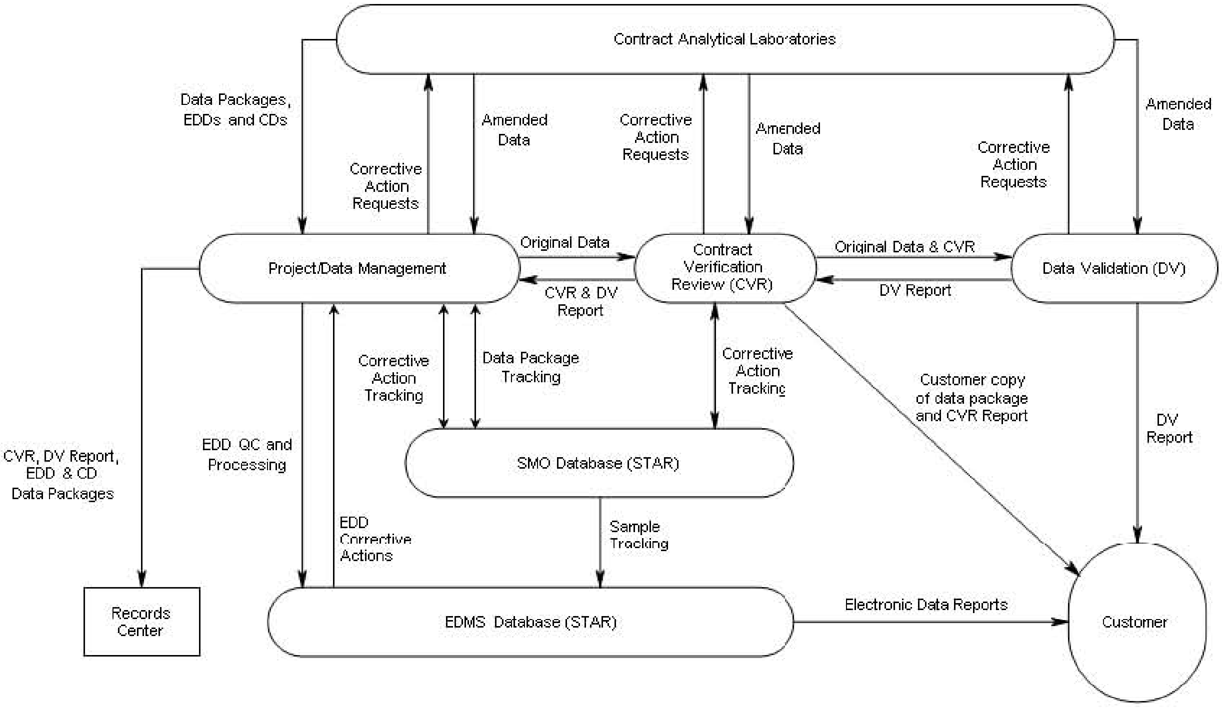
Attachment III

Sample Management Office
Sample/Financial Data Tracking Process



Attachment IV

Sample Management Office
Analytical Data Process



SANDIA NATIONAL LABORATORIES
LONG TERM STEWARDSHIP DEPARTMENT (4142)

SOIL MOISTURE DETERMINATION AT THE MIXED WASTE LANDFILL UTILIZING NEUTRON LOGGING

FIELD OPERATING PROCEDURE

FOP 10-07

Revision 1

Approved: Robert Ziock **Date:** 3/25/13
Robert Ziock, Author

Approved: Mike Mitchell **Date:** 3/25/13
Mike ~~Mitchell~~, Project Lead

Approved: Donald Schofield **Date:** 3/25/13
Donald Schofield, Field Support Operations Team Lead

Approved: **Date:** 4/1/2013
Steve Farmer, Radiological Protection

Approved: Pamela Puissant **Date:** 4/22/13
Pamela Puissant, Manager

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: 4/22/13

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), department home page.

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Revision History

Revision	Effective Date	Summary of Changes
Rev 0	2/28/2011	New Document
Rev 1	4/22/2013	Review cycle changed from 2 to 3 years. Updated to reflect the department's current formatting requirements for a FOP. Removed the following attachments: On-the-Job Training, Authorized Users List, and CPN 503 DR Hydroprobe® Moisture gauge Operating Manual. Updates were made to the following appendices: Radiological Hazardous Materials Summary; Neutron Probe (Source) Checklist for Shipping & Receiving; Mixed Waste Landfill Neutron Count Log Form.

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ACRONYMS AND ABBREVIATIONS

AOP	administrative operating procedure
cm	centimeter
CPN Probe	CPN 503 DR Hydroprobe® Moisture Depth Gauge
EDMS	Environmental Data Management System
EOC	Emergency Operations Center
ES&H	Environment, Safety and Health
ET Cover	evapotranspirative vegetative soil cover
FOP	field operating procedure
ft	foot (feet)
LTMMP MWL	Long-Term Maintenance and Monitoring Plan for the Mixed Waste Landfill
m	meter
mrem/hr	millirem per hour
MWL	Mixed Waste Landfill
NMED	New Mexico Environment Department
OJT	on-the-job training
OSHA	Occupational Safety and Health Administration
PHS	primary hazard screening
RCT	Radiological Control Technician
RPPM	Radiological Protection Procedures Manual
SNL/NM	Sandia National Laboratories/New Mexico
THA	task hazard analysis
TWD	technical work document
VZ	vadose zone

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1.0 PURPOSE, SCOPE, AND OWNERSHIP

The Mixed Waste Landfill (MWL) is located at Sandia National Laboratories/New Mexico (SNL/NM) Technical Area III. The MWL is a 2.6-acre site that was established in 1959 as a disposal area for low-level radioactive and mixed waste generated by SNL/NM research facilities. The MWL accepted low-level radioactive and minor amounts of mixed waste from March 1959 through December 1988. Approximately 100,000 cubic feet of low-level radioactive and mixed waste containing approximately 6,300 curies (at the time of disposal) of activity were disposed of at the MWL.

Purpose The purpose of this Field Operating Procedure (FOP) is to provide guidelines and procedures for use of the CPN503 DR Hydroprobe® Moisture Gauge (CPN probe) at the MWL. The CPN probe counts neutrons that will be used to determine soil moisture content beneath the MWL evapotranspirative vegetative soil cover (ET Cover). Neutron counts are correlated to moisture values by use of a correlation formula developed in the *Neutron-Probe Calibration Project at the Infiltration Pilot Site* (SNL/NM August 2001). Monitoring will be performed at the MWL to document levels of soil moisture in the vadose zone. This data will be used to evaluate the effectiveness of the MWL ET Cover as specified in the *Long-Term Maintenance and Monitoring Plan for the Mixed Waste Landfill* (LTMMP MWL) (SNL/NM March 2012).

Scope This FOP is applicable to all SNL employees and contractors who perform neutron logging activities at the Technical Area III MWL using a CPN probe. The work does not require a Radiological Work Permit and it will not affect other organizations.

Ownership The Long Term Stewardship Department is responsible for development, approval, distribution, revision, and control of this procedure.

2.0 RESPONSIBLE ORGANIZATION AND INDIVIDUALS

Responsible Organizations

The Long Term Stewardship Department is responsible for the following:

- Completion and documentation of soil moisture monitoring of the MWL vadose zone as outlined in the LTMMP MWL (SNL/NM March 2012).
- Performing the work in accordance with the Environment, Safety, and Health (ES&H) Radiological Protection Procedures Manual (RPPM) which implements the requirements of Corporate Procedure ESH100.2.RAD.1, Implement Radiation Protection Procedures.
- Storing and controlling access and tracking for the CPN probe.

Radiological Protection Department is responsible for providing the following:

- Providing guidance and support to Long Term Stewardship Department operation's involving radiological materials.

Responsible Individuals

The **Department Manager** is responsible for the following:

- Providing programmatic guidance leading to the development of this FOP.
- Performing assessments of MWL operations to ensure compliance to the LTMMP MWL and the ES&H RPPM.
- Reviewing and recommending approval of the procedure.
- Approving an Activity Level Work Evaluation Form (EP 2009-ALW) for activities described in this FOP as required by administrative operating procedure (AOP) AOP 09-10, *Work Planning and Control*.

The **Project Lead** (Job Coordinator equivalent in the ES&H RPPM) for vadose zone monitoring at the MWL is responsible for the following:

- Providing overall coordination and management of the monitoring activities.
- Reviewing and reporting soil moisture data.
- Transmitting soil moisture data to the Long Term Stewardship Database Administrator for inclusion in the Environmental Data Management System (EDMS).
- Reviewing and transmitting documentation forms to the Customer Funded Records Center and the Corrective Action Management Unit Administrative Trailer (Building 6920E).
- Reviewing, revising, and maintaining technical work documents (TWDs).
- Reviewing and recommending approval of this procedure.

The **Field Support Operations Team Lead** (Job Coordinator equivalent in the ES&H RPPM) is responsible for the following:

- Coordinating with the Project Lead and Field Technicians regarding monitoring activities.
- Supervising the Field Technicians.
- Reviewing training requirements for Field Technicians.
- Assigning qualified Field Technicians to conduct the activities described in this procedure.
- On-the-job training (OJT), as necessary, for new personnel performing field activities. Document training by completing on OJT Form (EP 2009-OJT).
- Providing Field Technicians with necessary equipment to conduct field work.

- Notifying the Project Lead of unusual field conditions, wells requiring maintenance, or breach of well security.
- Reviewing, revising, and maintaining technical work documents.

The **Field Technician** (Radiological Worker equivalent in the ES&H RPPM) is responsible for the following:

- Completing an Activity Level Work Evaluation Form (EP 2009-ALW) as required by AOP 09-10, *Work Planning and Control*.
- Notifying the site Radiological Control Technicians (RCTs) in SNL/NM Radiation Protection prior to using the CPN probe.
- Operating the CPN probe to obtain soil moisture data.
- Completing and reviewing field documentation forms.
- Transmitting field documentation forms to the Project Lead.
- Informing the Field Support Operations Team Lead of monitoring locations requiring maintenance or if the monitoring location has been compromised (e.g., lock has been removed, damage to protective enclosure or casing).
- Annual calibration of the CPN probe.
- Maintaining the equipment.
- Keeping training current.
- Providing recommendations for revisions to this procedure (if necessary).

The **RCT** is responsible for the following:

- Performing monthly radiation surveys of storage area for the CPN probe.
- Performing semi-annual leak surveys of the CPN probe.
- Performing shipping surveys when the CPN probe is sent to manufacturer for calibration and/or maintenance, and when it is returned to SNL/NM.
- Providing radiological protection support to the field activity in accordance with the ES&H RPPM.

The **Database Administrator** is responsible for the following:

- Importing data into EDMS.
- Maintaining the database.
- Identifying, recommending, and implementing improvements to the database.

The **Source Custodian** and **Alternate Source Custodian** responsibilities are detailed in the ES&H RPPM, Chapter 9, Control of Accountable Sealed Radioactive Sources, Source Custodian Section 9.3.3.

The **ES&H Coordinator** is responsible for assisting the staff and management in ES&H performance and compliance.

3.0 TRAINING AND MONITORING QUALIFICATIONS

Personnel conducting field activities shall complete the following:

3.1 Training

- Read SNL/NM Corporate Policy ESH100 Environment Safety & Health.
- Complete required department training and training identified in the primary hazard screening (PHS) results.
- Read PHS SNL06A00497, *Vadose Zone Monitoring at the Mixed Waste Landfill*.
- Read applicable sections of the LTMMP MWL (SNL/NM 2012).
- Pre-job briefing required by the ES&H RPPM. (Note: Signing the Authorized Users List (EP2009-AUL) suffices for the pre-job briefing required by the ES&H RPPM.)
- Read *CPN 503 DR Hydroprobe® Moisture Gauge Operating Manual*.
- Familiarity with sections in the ES&H RPPM that pertain to sealed and controlled radioactive sources.
- OJT, as necessary, for new personnel performing field activities. Document training by completing on OJT Form (EP 2009-OJT).
- Complete training courses listed in Table 1.
- 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.

Table 1 - Training Course List

Course Code	Course Title
CHM100	Chemical Safety
CHM103	Site Specific Chemical Safety
ELC105	Basic Electrical Safety (> 50 volts)
ENV100	Occupational Safety and Health Administration (OSHA) Health & Safety Basic Training - General Worker (40 HR)
ENV103	OSHA Health & Safety Training Refresher (8 HR)
ENV112	Hazardous Waste & Environmental Management Training
ESH100	ES&H Awareness
MCH200	Hand and Power Tool Safety
MED102	Standard First Aid
MED104	Heartsaver CPR
OTS101	Occupational Thermal Stress
PPE106	Personal Protective Equipment Training
RAD210	Radiological Worker I Training

3.2 Monitoring

Personnel using the CPN probe are responsible for obtaining and wearing properly coded (neutron radiation) dosimeters during all operations. They are also responsible for returning the dosimeter to their Department Manager on the assigned dates.

4.0 HEALTH AND SAFETY

A task hazard analysis (THA) has been performed on the activities described in this FOP. The THA was performed in conjunction with the latest version of the PHS SNL06A00497. 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. The THA lists control measures that will be used to mitigate the potential hazards. 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 the SNL Integrated Safety Management System initiative.

An exposure assessment survey for field activities was performed by an SNL/NM industrial hygienist. The exposure assessment survey report concluded that the potential for exposure to health hazards has been categorized as well-controlled; therefore acceptable.

An Activity Level Work Evaluation Form was completed and approved by the Department Manager as required by AOP 09-10. The hazards and controls to minimize the hazards are listed in Table 2.

A tailgate safety and emergency response briefing shall be conducted before the start of field activities. A Tailgate Safety Meeting Form (Appendix A) shall be completed at time of aforementioned briefing.

Table 2 – Task Hazard Analysis

Potential Hazard	Hazard Rating	Control
Radiological (see Section 4.1 for additional information) • External Radiation • Leaking Source • Contaminated materials or soil	Medium	• Personnel must be trained on the radiological hazards associated with the use of the instrument and are required to wear their radiation dosimetry during use. • The instrument (source) must be leak tested semi-annually by SNL Radiation Protection. • Work is performed outside site boundary and an ET Cover has been installed over the former disposal areas (minimum average thickness of 5.37 feet [ft]); therefore personnel will not come into contact with contaminated materials or soil. • Monitoring wells have enclosed steel casing which prevents the monitoring equipment from coming into contact with contaminated materials or soil.
Chemical	Low	• Work is performed outside site boundary and an ET Cover has been installed over the former disposal areas (minimum average thickness of 5.37 ft); therefore personnel will not come into contact with contaminated materials or soil. • Monitoring wells have enclosed steel casing which prevents the monitoring equipment from coming into contact with contaminated materials or soil.
Physical • Weather/Heat Stress & Cold Stress • Sunburn • Mechanical hazards (cable winch) • Strains, and lifting hazards • Slips, trips, falls	Low	• Monitoring activities are not physically demanding. • First Aid kit is located in sampling vehicle. • Personnel should seek shelter if lightning is observed within 5 miles (25 seconds from time of flash to thunder) or the Emergency Operations Center (EOC) issues a lightning warning (via EOC pager). Workers will be trained on heat & cold stress, and sunburn hazards. Sunscreen will be provided. • Dangling lanyards will not be worn when operating manual cable winch. • Leather work gloves will be worn when handling the cable from the winch. • Proper lifting techniques will be reinforced. • Holes will be filled or covered to eliminate slip, trip hazards.
Biological • Snakes, Rodents, Insects	Low	• Care will be taken to observe that the well casings pose a potential for insects and other animals. • Monitoring well areas will be kept clean and places of refuge for biological hazards minimized.
Fire	Low	• Fire extinguishers will be located in mobile equipment.

4.1 Radiation Hazard

The CPN probe contains a 50.0 millicurie Am241/Be neutron source. Radiation levels provided by the manufacturer are:

30 centimeters (cm): 0.1 millirem per hour (mrem/hr) gamma, 1.8 mrem/hr neutron

Contact: 0.5 mrem/hr gamma, 1.7 mrem/hr neutron
Other: up to 30 mrem/hr neutron on contact if unshielded

Detailed ES&H RPPM requirements for use and storage of the CPN probe are listed below:

Engineering Controls: None
Administrative Controls:
- Pre-Job Briefing shall be performed.
- Technical Work Documents (TWDs) (FOP, PHS, and THA).

Hold Points: None
Void Points: Contamination greater than ES&H RPPM Attachment 6-1 Radioactive Contamination Limits of 20 disintegrations per minute/100 cm^2 and radiation levels greater than 5 mrem/hr at 30 cm (gamma + neutron).
Alarming Equipment (or other special equipment): None
RCT Coverage: Intermittent
RCT required surveys:
- Semi-annual leak test (cycle group April and October)
- Monthly routine survey of storage area
- Transportation surveys when shipping CPN probe to manufacturer.
- Job coverage surveys as needed

Source Custodian required inventory: Semi-annual (cycle group April and October)
Radiological Posting:
- Radioactive Materials Area
- Controlled Area

Frisking Requirements: None
TWD (this FOP) Sign-In Requirements: Initial

For more information on the CPN probe see Radiological Hazardous Materials Summary (Appendix B).

Extreme care shall be taken to ensure safe operation in accordance with this procedure and to keep radiation exposure as low as reasonably achievable (ALARA). If the CPN probe becomes stuck in an access tube or borehole shut down work immediately , proceed as directed in section 4.2 Work Shutdown.

4.2 Work Shutdown

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 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 Control*. The Department Manager shall sign the completed form prior to the restart of work.

In the event that work is stopped due to:

- The CPN probe becoming lodged in an access tube, call Radiation Protection and inform them of the problem. An RCT will be dispatched to the site. Do not attempt to retrieve the probe in any manner that could possibly damage the probe or expose the source.
- The CPN probe sustaining extensive damage, maintain a safe distance from the probe (10 meters [m] or 33 ft). Immediately notify Radiation Protection. An RCT will be dispatched to the site.

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 Control*. The Department Manager shall sign the completed form prior to the restart of work.

5.0 DATA QUALITY OBJECTIVES

Refer to the LTMMP MWL (SNL/NM March 2012) for data quality objectives.

6.0 EQUIPMENT AND MATERIALS

6.1 Personal Protective Equipment

Personal protective equipment includes the following:

- safety boots
- safety glasses
- leather gloves (when handling CPN interface cable)

6.2 Neutron Logging Equipment and Materials

The CPN 503DR system consists of the following (see Appendix C for photos):

- CPN probe
- CPN probe shielded control box
- Interface cable (12 ft length used to connect CPN probe to shielded control box for standard count)
- Instrument suitcase
- CPN cable reel mounted in vehicle (spooled with 375 ft of CPN interface cable)
- Battery charger

Additional equipment and materials includes the following:

- Logbook
- Pulley fixture to be mounted to the access tube.
- Power source (12 volt DC to AC vehicle power inverter mounted in vehicle)
- Key to unlock padlock on building ERFO2 (obtain key from the Project Lead)
- Key to unlock padlocks on access tube protective casings (obtain key from Project Lead)

7.0 FIELD PROCEDURES

7.1 CPN Probe Handling Requirements

The CPN probe is stored in building ERFO2. It can only be used with the permission of the Source Custodian. The designated support RCT must also be notified prior to using the CPN probe. The Field Technician must complete the following information on the

Sign-Out/Sign-In Form (Appendix D) when using the CPN probe:

- Field Technician name
- Source Custodian contacted prior to us
- RCT contacted prior to use
- Reason for using CPN probe
- CPN probe Source ID
- Sign-out date and time
- Sign-in date and time

The probe should always remain in the shielded control box unless in use. When the probe is outside the shielded control box, use the shielding blanket to cover the probe whenever possible. The operator should minimize their time near the probe. A distance of at least a 1-m (3.3-ft) should be maintained. Do not leave the probe unattended, except when it is locked in building ERFO2 for storage. The door to ERFO2 is clearly marked with a magenta and yellow placard labeled “Caution-Radioactive Materials.” The CPN shielded control box and its storage case are labeled with the appropriate radioactive material warnings. These labels must be replaced immediately if damaged, obscured, or removed. Contact Radiation Protection to assist in the replacement of signs and/or labels. Personnel not performing the monitoring, but in the immediate area, need to be notified of the radiation hazard. Only a government vehicle may be used to transport the CPN probe.

A leak test survey is performed on the probe by radiation protection personnel semi-annually (cycle group April and October). A source inventory is performed by the Source Custodian semi-annually (cycle group April and October). Both are requirements listed in the ES&H RPPM. A notice should be issued by the Device and Source Registrar indicating when the survey and inventory are due. In addition, a Source Status Label (Appendix E) is attached to the CPN shielded control box and its storage case stating when the leak test survey and semi-annual inventory were last performed and when they need to be updated.

If the CPN probe needs to be sent to the manufacturer for calibration and/or maintenance, follow the requirements listed in the ES&H RPPM. The Neutron Probe (Source) Checklist for Shipping & Receiving is provided in Appendix F that gives detailed directions for shipping and receiving. Update the Source Status Label (Appendix E) to "Active" or "Storage" as necessary.

7.2 Standard Count

Charge the battery in the CPN probe shielded control box prior to use. A standard count will be taken each day the CPN probe is used (see page 21 of *CPN 503 DR Hydroprobe® Moisture Gauge Operating Manual*). Take five standard counts if the CPN probe is new, has been repaired, or the probe has not been used for six months. The standard count measurement is taken with the probe in the shielded control box. Always place the probe in the same location when taking standard counts to ensure consistency.

1. Place the CPN probe storage case on a sturdy, level surface (level ground is fine) at least 3 m (10 ft) from a hydrogen source or any large vertical surface, and at least 10 m (33 ft) from any other radioactive source.
2. Place the shielded control box on top of the standard count plate (a plate on top of the probe storage case). Leave the probe in the shielded control box.
3. Connect the CPN interface cable (10-ft length) between the probe and the shielded control box.
4. Press the "CLEAR" key to display "READY."
5. Press the "STD" key to get into the standard count menu. This will display the current standard count.
6. Press the "STEP" key. This will display the previous standard count.
7. Press the "STEP" key. This will display the current Chi squared (χ^2) value.
8. Press the "STEP" key. This will display "New Standard?"
9. Press "Enter."
10. Step back to a safe distance of at least 10 ft.

The shielded control box will beep after pressing “ENTER.” This indicates it has begun counting to determine a new standard count. The counting will continue for approximately 4 minutes. The shielded control box will beep three times when the standard count is completed and then display the new standard count value.

11. Press the “ENTER” key. This will save the new standard count value and display the “READY” prompt.

The new standard count is acceptable if the new χ^2 value is between 0.75 and 1.25. To display the new standard count and new χ^2 value repeat steps 5 through 7. Record the new standard count and the new χ^2 value on the <u>Mixed Waste Landfill Neutron Count Log Form</u> (Appendix G) if $\chi2$ is within the acceptable range. If the new χ^2 is not between 0.75 and 1.75, begin with step 4 again. Otherwise continue with step 12. (Refer to the discussion on statistics and other standard count information contained in the CPN 503 DR Hydroprobe® Moisture Gauge Operating Manual).

12. Press the “STEP” key. This will display “New Standard?”
13. Press “CLEAR.”
14. This will display the “READY” prompt.

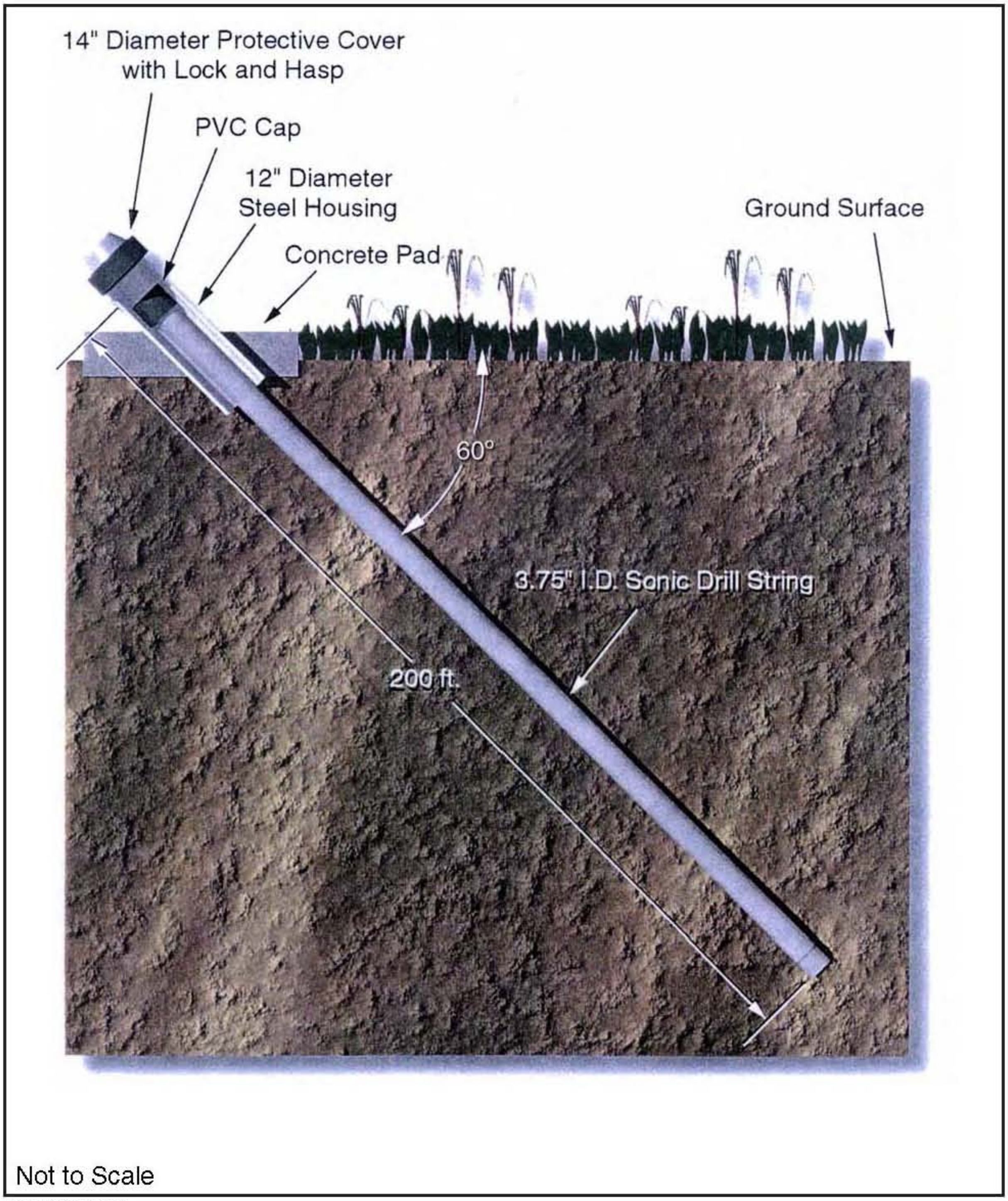
After taking the standard count verify that the sampling time is set for 30 seconds. Press the “TIME” key. If the display shows “30,” press “ENTER” and the probe is ready for field measurements. If the display shows something other than “30,” use the “STEP” key to scroll through the time choices until “30” is displayed. Press “ENTER,” and the probe is ready for field measurements.

7.3 Neutron Logging of Access Tubes

The soil-moisture monitoring system is comprised of three access tubes drilled on a 30-degree angle from vertical to a depth of 200 linear feet and a vertical depth of 173 feet below ground surface. The access tubes are identified as vadose zone (VZ)-1, VZ-2, and VZ-3. Each access tube is cased with 4.5-inch diameter steel casing. The access tubes are open to the soil in the bottom (no end cap). Figure 1 presents a schematic of an access tube. To log neutron counts:

1. Notify the designated support RCT that the CPN probe will be used.
2. Take the standard count (refer to Section 6.2).
3. Position the CPN cable reel in line with the access tube casing.
4. Open lock and remove casing cap.
5. Attach unistrut pulley fixture to top side of casing.
6. Attach the CPN cable to probe while probe still in shielded control box.
7. Place shielded control box directly next to access tube casing.
8. Remove the probe from the shielded control box. Thread the probe through the unistrut pulley assembly and into the access tube casing.

Figure 1 – Soil Moisture Monitoring Access Tube



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9. Slowly lower the CPN probe into the access tube until the bottom of the CPN probe is even with the ground level. Set the cable reel footage counter to zero.
10. Place shielded control box next to CPN cable reel and attach cable coming from cable reel hub to the shielded control box.
11. Reel out the CPN probe to the first monitoring depth.
12. Press the "COUNT" button on the shielded control box. A neutron count value will be displayed after 30 seconds.
13. Record neutron count value on Mixed Waste Landfill Neutron Count Log Form (Appendix G).
14. Continue to move the probe to each predetermined location in the access tube to collect neutron counts.

When all sample data have been collected:

15. Disconnect cable from shielded control box.
16. Place shielded control box directly next to access tube casing.
17. Remove probe from access tube casing and immediately place into shielded control box.
18. Disconnect cable from probe.
19. Close and lock the casing cap.

Repeat this procedure for the remaining access tubes. Appendix C provides photos of the equipment setup.

8.0 WASTE MANAGEMENT

No waste is generated from neutron logging activities.

9.0 DATA MANAGEMENT

9.1 Data Collection Locations and Frequency

Neutron logging data is collected from three locations (VZ-1, VZ-2, and VZ-3). There are sixty monitoring points (depths) for each of the VZ locations. See the Mixed Waste Landfill Neutron Count Log Form (Appendix G) for monitoring depths. The frequency at which the data is collected is specified in the LTMMP MWL.

9.2 Data Submittal Process

The steps of the data flow process and the personnel associated with each step are defined below.

Field Technician

1. As the neutron count data is collected and recorded on the Mixed Waste Landfill Neutron Count Log Form (Appendix G), it is compared to the previous monitoring result values to see if there are any anomalies.
2. Anomalies are noted and the Mixed Waste Landfill Neutron Count Log Form (Appendix G) is submitted to the Project Lead.

Project Lead

1. The Project Lead enters the neutron count data into a Microsoft® Excel spreadsheet that uses a previously determined correlation equation to convert neutron counts to soil moisture content (percent water content by volume).
2. Neutron count and soil moisture data are submitted to the Database Administrator after review by the Project Lead.

Database Administrator

The Database Administrator imports the data received from the Project Lead into EDMS.

10.0 REFERENCES

InstroTek, Inc. (formerly CPN Company). CPN 503 DR Hydroprobe® Moisture Gauge Operating Manual, 4057 Port Chicago Hwy STE 100, Concord, California. http://instrotek.com/

Sandia National Laboratories, Corporate Policy ESH100 Environment Safety & Health (current version), Sandia National Laboratories, New Mexico.

Sandia National Laboratories, "Neutron-Probe Calibration Project, Infiltration Pilot Site, Mixed Waste Landfill," Sandia National Laboratories, Albuquerque, New Mexico (2001).

Sandia National Laboratories, "Long-Term Monitoring and Maintenance Plan for the Mixed Waste Landfill," Sandia National Laboratories, Albuquerque, New Mexico (2012).

Sandia National Laboratories, "Work Planning and Control," AOP 09-10 (current version), Sandia National Laboratories, Albuquerque, New Mexico.

Sandia National Laboratories, MN471016, Radiological Protection Procedures Manual (current version), Sandia National Laboratories, Albuquerque, New Mexico.

Sandia National Laboratories Primary Hazard Screening, SNL06A00497 (current version), “Vadose Zone Monitoring at the Mixed Waste Landfill.” Sandia National Laboratories, Albuquerque, New Mexico.

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APPENDIX A
Tailgate Safety Meeting Form

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Tailgate Safety Meeting Form

Dept: ________ Facility: MWL Date: _____________ Time: _________

Activities: Soil moisture monitoring using CPN503DR Hydroprobe.

(Anyone has the right to cease field activities for safety concerns. The buddy system will be used when performing field work.)

Weather Conditions:
Temp: _____°F Wind Speed: _________MPH Humidity: _____% Wind Chill: ______°F

☐ Wear safety boots		☐ Wear leather gloves
☐ Wear safety glasses		☐ Wear sun screen
☐ Be aware of biohazards (snakes, spiders, etc.)		☐ Wear communication device (cell phone, EOC pager)
☐ Be aware of slips, trips, and falls		☐ Using safe lifting practices were discussed.
☐ Be aware of pinch points on winch		☐ Be aware of environmental conditions (heat/cold stress)
Does anyone have any weight restrictions on lifting? Circle **YES** or **NO**. If answered **YES** explain.		
☐ Practice ALARA		☐ Notify RCT when using neutron probe

ATTENDEES

Printed Name	Signature
Printed Name	Signature
Printed Name	Signature
Printed Name	Signature
Printed Name	Signature
Printed Name	Signature

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), department home page.

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APPENDIX B
Radiological Hazardous Materials Summary

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RADIOLOGICAL HAZARDOUS MATERIALS SUMMARY

General Information

Type of device: – Neutron Moisture Probe
Manufacturer: – CPN International, Inc.
Model Number: – CPN 503DR
Serial Number: – #H30039459 (primary instrument), #H300605758 (backup instrument)
Operating parameters: – 6.5 mA Average
1.85 gigabecquerel (50 millicurie) Americium-241:Beryllium
Type of radiation: – Gamma, Neutron
Sandia Device and Source Registrar ID#: – RS02625 (primary instrument), RS00661 (backup instrument)
Location of device: – TA-III, Building ERFO2

The manufacturer’s encapsulation of the source is a double sealed capsule CPN-131.
The manufacturer’s shielding of the source is provided by silicon-base paraffin.

Source Custodian: Robert Ziock, Dept. 4142, 845-0485
Alternate Custodian: none

Radiation Levels (provided by manufacturer)

30 cm: 0.1 mrem/hr gamma, 1.8 mrem/hr neutron
Contact: 0.5 mrem/hr gamma, 1.7 mrem/hr neutron
Other: up to 30 mrem/hr neutron on contact if unshielded

Dosimetry

A TLD with a neutron code of 20 is required for personnel operating the CPN probe.

Training and Technical Work Documents

Operators of the CPN probe shall complete the following training:

- 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.
- OJT as necessary, for new personnel performing field activities. Document training by completing on OJT Form (EP 2009-OJT).
- Read *CPN 503 DR Hydroprobe® Moisture Gauge Operating Manual*.
- OSHA 40-hr Hazardous Waste Operations Training.
- OSHA 8-hr Hazardous Waste Operations Training Refresher

RADIOLOGICAL HAZARDOUS MATERIALS SUMMARY (continued)

- Read PHS SNL06A00497, *Vadose Zone Monitoring at the Mixed Waste Landfill.*
- RAD210 Radiological Worker 1.
- Familiarity with sections in the ES&H RPPM that pertain to sealed and controlled radioactive sources.

ALARA protocol will be performed by the field personnel when using this instrument.

Postings

Postings at storage location include: “Controlled Area” and “Radioactive Materials”

Users of the instrument will be responsible for administrative controls while the probe is in use or transport.

Surveys & Inventory

A leak survey shall be performed on the CPN probe semi-annually (cycle group April and October) by an RCT.

A leak test survey shall also be performed on the CPN probe prior to transportation by the Packaging and Transportation group from the ERFO2 storage building. This will occur only when the CPN probe is sent to the manufacturer for calibration/maintenance. Upon return to the ERFO2 storage building from the manufacturer and prior to use of the CPN probe by a Field Technician, an additional leak test survey must be performed.

> **Note**: Anytime the CPN probe is shipped to the manufacturer, the status of the source must be change from “**Active**” to “**Storage**” status prior to movement from the ERFO2 storage building. This is done via the “Device and Radioactive Source Tracking System” website at: https://webprod2.sandia.gov/dvs/. Upon return from the manufacturer and prior to use of the CPN probe by a Field Technician, the status of the source must be changed back to “**Active**” status.

Monthly routine surveys of the ERFO2 storage building are performed by an RCT.

Inventory of the CPN probe is performed semi-annually (cycle group April and October) by the Source Custodian.

RADIOLOGICAL HAZARDOUS MATERIALS SUMMARY: (concluded)

Transport

- Only within TA-III by authorized field personnel. Any other transportation will be done by the Packaging and Transportation group.
- Instrument will be transported in the manufacturer's hard shell suitcase. The suitcase will be secured in the vehicle using tie-downs.
- A "Caution Radioactive Material" label must be affixed to the outside of the suitcase.
- If the CPN probe is used in the field for more than five days (without daily storage in ERFO2) or is transferred off-site, update the source location in the Radiation Protection Source Database.

Operational As Low As Reasonably Achievable (ALARA) Screen

The Operational ALARA Screen shall be performed by evaluating the following conditions. If any of the conditions apply to the radiological work an Operational ALARA Review shall be performed. [10 Code of Federal Regulations 835.1003(b)]

- Will the highest individual dose of >100 mrem total effective dose be expected to complete the work? No.
- Will the collective dose of >500 person-mrem total effective dose be expected to complete the work? No.
- Will airborne radioactivity in the accessible work area be expected to routinely meet or exceed the criteria for an airborne radioactivity area? No airborne radioactivity.
- Will removable contamination in the accessible work area be expected to routinely meet or exceed the criteria for a high contamination area? No removable contamination.
- Will hot particles be expected in the accessible work area? No hot particles.
- Will general area dose rates in the accessible work area be expected to routinely meet or exceed the criteria for a high or very high radiation area? No.
- Are dose rates of >50 µrem/hr expected in occupied areas for a period >1 week? No.

An Operational ALARA Review is not required based on the answers to the above questions.

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APPENDIX C

Photographs of Equipment Setup

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CPN probe/shielded control box and instrument case (probe contained in shielding)

CPN probe/shielded control box and instrument case (probe removed from shielding)

Cable reel used to lower CPN probe into the VZ access tubes

Equipment setup at a VZ monitoring location

APPENDIX D

CPN Probe Sign-Out/Sign-In Form

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CPN Probe Sign-Out/Sign-In Form

Field Technician Name	Field Technician Signature	Source Custodian Contacted?	RCT Contacted?	CPN Probe Used	Purpose	Sign Out Date/Time	Sign In Date/Time
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			
		☐ Yes	☐ Yes	☐ RS00661 ☐ RS02625			

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APPENDIX E
Source Status Label

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Source Status Label

Example:

Source ID #: RS00661
Status: ACTIVE
Leak Test last performed: 4/11/05
Leak Test due by: 10/11/05
Semi-Annual Inventory Performed: 4/27/05
Semi-Annual Inventory due by: 10/27/05

Replace with "STORAGE" status tag if the unit is sent in for calibration/maintenance. Upon return, immediately have a leak test survey performed to upgrade the status to "ACTIVE" and indicate status on a new tag. Notify Device & Source Registrar of any status or storage changes: 844-3415

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APPENDIX F

Neutron Probe (Source) Checklist for Shipping & Receiving

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Neutron Probe (Source) Checklist for Shipping & Receiving
(To be completed by Source Custodian only)

Indicate Source Registrar ID # of neutron probe instrument to be shipped to manufacturer for calibration and/or repairs:

☐ RS00661 ☐ RS02625

Current status of the source indicated on instrument and instrument storage case label. (It should indicate that the source is "Active")

☐ Active ☐ Storage

Notify a Radiological Control Technician (RCT) that the instrument will be sent offsite (to the manufacturer) for calibration and/or repairs and request a "Movement" and "Shipping" survey.

☐ RCT notified

☐ RCT performed "Movement" and "Shipping" survey. Date: __________________

☐ RCT informed shipper of the "Movement" and "Shipping" survey # _________________

Notify "Device and Source Registrar" by e-mail devsrc@sandia.gov that there will be an offsite transfer of the source to the manufacturer for calibration and/or repairs. Request a "**Change of Status**" from "**Active**" source to "**Storage**". Be sure to specify the Source Registrar ID #. For more information go to the "Device and Source Registrar" at https://webprod2.sandia.gov/dvs/

☐ Send e-mail to devsrc@sandia.gov indicating that the source will be transferred to the manufacturer for calibration and/or repairs and request a "**Change of Status**" from "**Active**" source to "**Storage**."

☐ Print a hardcopy of all e-mails to and from the "Device and Source Registrar" And keep with instrument's records.

☐ Date that "Device and Source Registrar" was notified: ___________________

Replace tag on the instrument and the instrument storage case indicating the status has been changed from "**Active**" to "**Storage**."

☐ Tag has been replaced. Date tag was replaced: ___________________

Complete shipping documentation (SHIPPER form) located at the following web address:

http://cfo.sandia.gov/logistics/Shipping/Page1WSF.htm

Neutron Probe (Source) Checklist for Shipping & Receiving (continued)

Attach all relevant documents electronically to the SHIPPER form as instructed. This includes:

☐ The "Information of Hazardous Material Shipments" form (provided on the SHIPPER website). Indicate on appropriate line the material to be shipped is CLASS 7-RADIOACTIVE MATERIALS.

☐ The supplemental Radioactive Shipment Information form (provided on the SHIPPER website). An RCT can help provide the information requested on the form.

☐ The "Movement" and "Shipping" survey that was performed by the RCT.

☐ The most recent "SPECIAL FORM" provided by the manufacturer (see SPECIAL FORM attached to this checklist).

☐ Copy of letter from the National Nuclear Security Administration Sandia Site Office to SNL Radiation Protection "Exemption of Department of Energy and its Prime Contractors from Nuclear Regulatory Commission Licensing Requirements" (see letter attached to this checklist).

☐ Copy of U.S. Department of Transportation (Pipeline and Hazardous Materials Safety Administration) International Certificate of Competent Authority Special Form Radioactive Materials Certificate USA/0632/S-96 (see certificate attached to this checklist).

☐ Copy of Radioactive Material License Number 1100-07 (see license attached to this checklist.

☐ Copy of the Safety Data Sheet for secondary nickel-cadmium sealed cells (see Safety Data Sheet attached to this checklist).

☐ A statement in the form of an e-mail from a Nuclear Criticality Safety Engineer (NCSE) that certifies the material remains below the DeMinimis requirements (see e-mail attached to this checklist).

Print a hardcopy of all the forms and keep with instrument's records.

☐ Forms printed.

Submit the SHIPPER form and all attachments.

☐ Date submitted: ____________________

Neutron Probe (Source) Checklist for Shipping & Receiving (concluded)

A Customer Service Request (CSR) Form must be completed after the Shipper is submitted. The CSR Form can be accessed at:

https://arsprod.sandia.gov/Logistics/LogisticsNM.asp

☐ CSR Form completed and submitted. Date submitted: ____________________

☐ Instrument was picked up by shippers for delivery to the manufacturer. Source Custodian signed shipping form indicating instrument pickup.

The following will be completed upon return of the instrument to Sandia National Laboratories:

☐ The Source Custodian was notified that the instrument has been returned from the manufacturer and a delivery to the Source Custodian is arranged.

☐ Source Custodian signed for instrument and returned it to Building ERFO2.
Date: _________________

☐ Shipping papers were filed with the instrument's records.

☐ Source Custodian immediately notifies the "Device Source Registrar" via e-mail that the instrument has been returned from the manufacturer to regular storage location at TA-III, Building ERFO2. Date notified: ___________

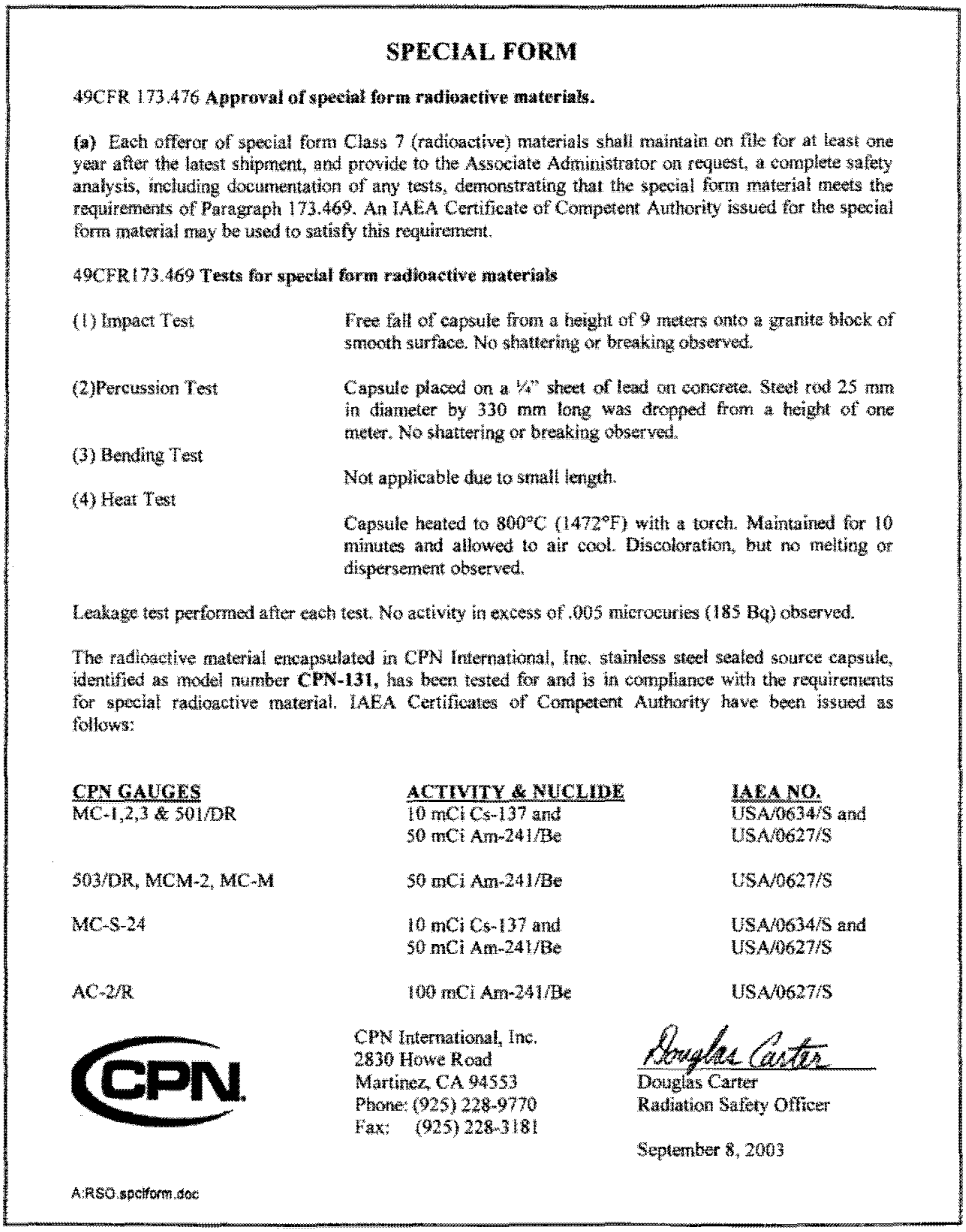
☐ Source Custodian schedules leak test with an RCT.
Date leak test performed: ___________

☐ Source Custodian e-mails "Device Source Registrar" a copy of the leak test results and request a "**Change of Status**" from "**Storage**" source to "**Active**" if the leak test indicates that the source is intact.

☐ Print a hardcopy of all e-mails to and from the "Device and Source Registrar" And keep with instrument's records.

Replace tag on the instrument and the instrument storage case indicating the status has been changed from "**Storage**" to "**Active**."

☐ Tag has been replaced. Date tag was replaced: __________________



SPECIAL FORM

49CFR 173.476 **Approval of special form radioactive materials.**

(a) Each offeror of special form Class 7 (radioactive) materials shall maintain on file for at least one year after the latest shipment, and provide to the Associate Administrator on request, a complete safety analysis, including documentation of any tests, demonstrating that the special form material meets the requirements of Paragraph 173.469. An IAEA Certificate of Competent Authority issued for the special form material may be used to satisfy this requirement.

49CFR173.469 **Tests for special form radioactive materials**

(1) Impact Test	Free fall of capsule from a height of 9 meters onto a granite block of smooth surface. No shattering or breaking observed.
(2)Percussion Test	Capsule placed on a ¼" sheet of lead on concrete. Steel rod 25 mm in diameter by 330 mm long was dropped from a height of one meter. No shattering or breaking observed.
(3) Bending Test	Not applicable due to small length.
(4) Heat Test	Capsule heated to 800°C (1472°F) with a torch. Maintained for 10 minutes and allowed to air cool. Discoloration, but no melting or dispersement observed.

Leakage test performed after each test. No activity in excess of .005 microcuries (185 Bq) observed.

The radioactive material encapsulated in CPN International, Inc. stainless steel sealed source capsule, identified as model number **CPN-131**, has been tested for and is in compliance with the requirements for special radioactive material. IAEA Certificates of Competent Authority have been issued as follows:

<u>CPN GAUGES</u>	<u>ACTIVITY & NUCLIDE</u>	<u>IAEA NO.</u>
MC-1,2,3 & 501/DR	10 mCi Cs-137 and 50 mCi Am-241/Be	USA/0634/S and USA/0627/S
503/DR, MCM-2, MC-M	50 mCi Am-241/Be	USA/0627/S
MC-S-24	10 mCi Cs-137 and 50 mCi Am-241/Be	USA/0634/S and USA/0627/S
AC-2/R	100 mCi Am-241/Be	USA/0627/S

CPN International, Inc.
2830 Howe Road
Martinez, CA 94553
Phone: (925) 228-9770
Fax: (925) 228-3181

Douglas Carter
Douglas Carter
Radiation Safety Officer

September 8, 2003

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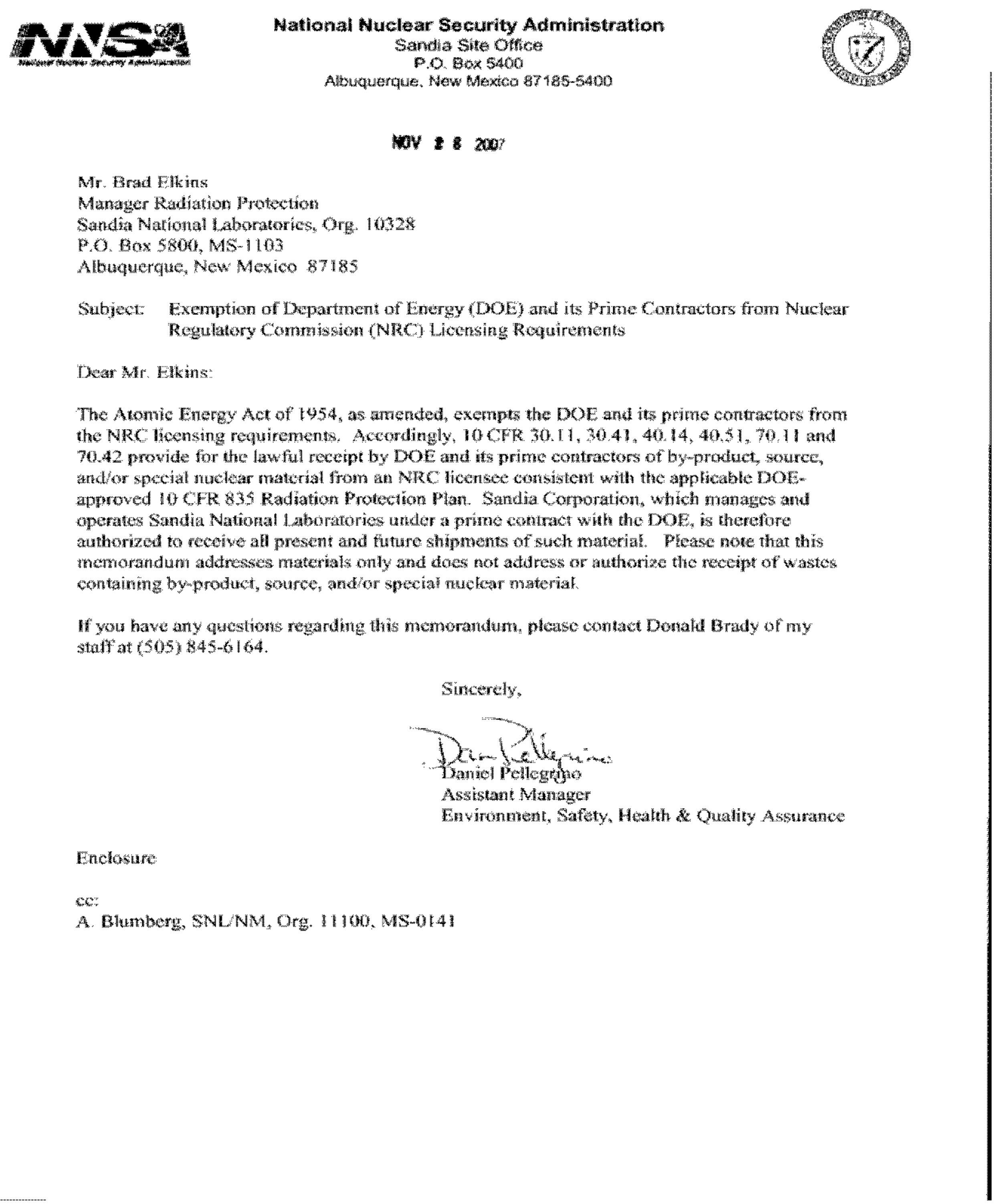


National Nuclear Security Administration
Sandia Site Office
P.O. Box 5400
Albuquerque, New Mexico 87185-5400

NOV 28 2007

Mr. Brad Elkins
Manager Radiation Protection
Sandia National Laboratories, Org. 10328
P.O. Box 5800, MS-1103
Albuquerque, New Mexico 87185

Subject: Exemption of Department of Energy (DOE) and its Prime Contractors from Nuclear Regulatory Commission (NRC) Licensing Requirements

Dear Mr. Elkins:

The Atomic Energy Act of 1954, as amended, exempts the DOE and its prime contractors from the NRC licensing requirements. Accordingly, 10 CFR 30.11, 30.41, 40.14, 40.51, 70.11 and 70.42 provide for the lawful receipt by DOE and its prime contractors of by-product, source, and/or special nuclear material from an NRC licensee consistent with the applicable DOE-approved 10 CFR 835 Radiation Protection Plan. Sandia Corporation, which manages and operates Sandia National Laboratories under a prime contract with the DOE, is therefore authorized to receive all present and future shipments of such material. Please note that this memorandum addresses materials only and does not address or authorize the receipt of wastes containing by-product, source, and/or special nuclear material.

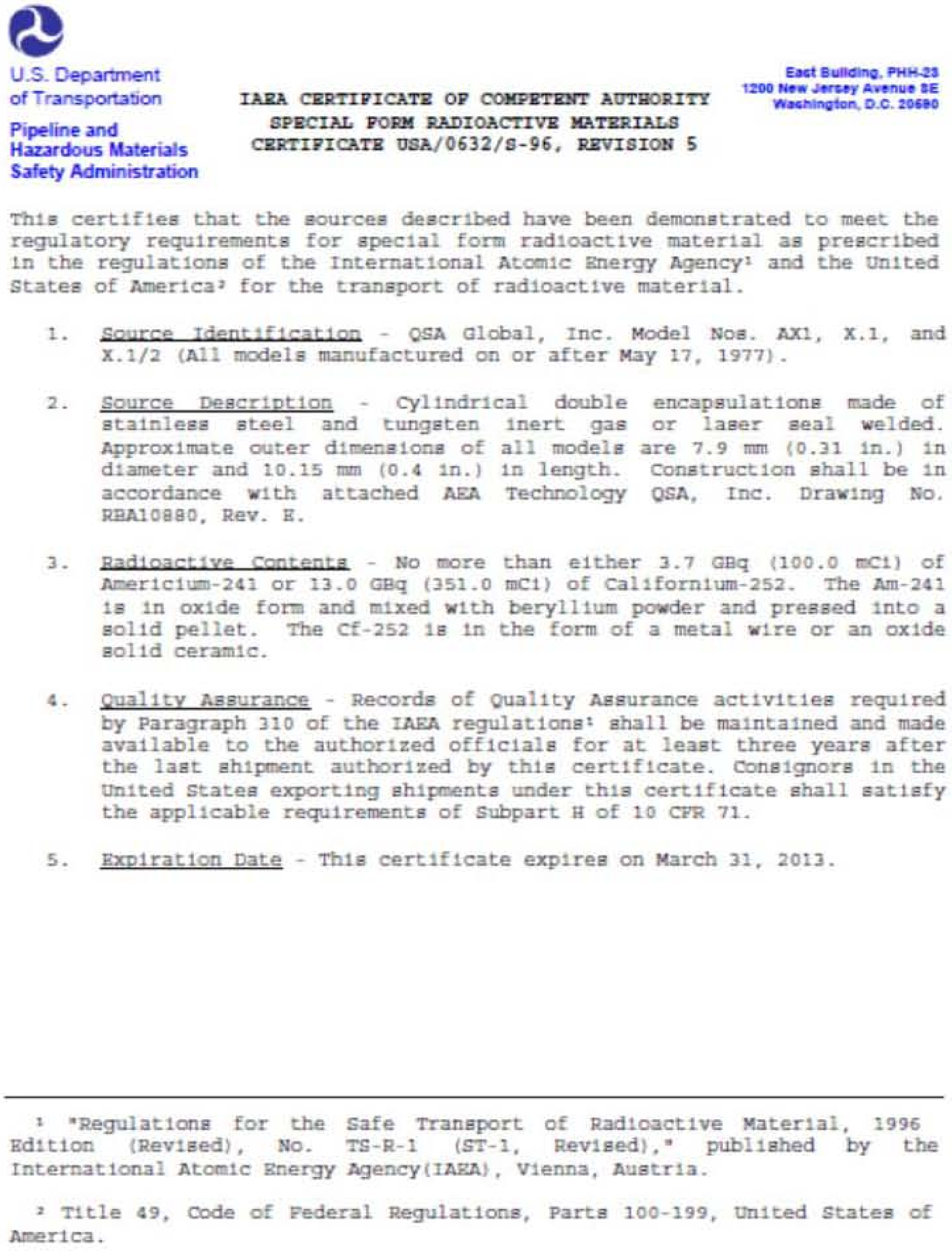
If you have any questions regarding this memorandum, please contact Donald Brady of my staff at (505) 845-6164.

Sincerely,

Daniel Pellegrino
Assistant Manager
Environment, Safety, Health & Quality Assurance

Enclosure

cc:
A. Blumberg, SNL/NM, Org. 11100, MS-0141



U.S. Department
of Transportation
**Pipeline and
Hazardous Materials
Safety Administration**

**East Building, PHH-23
1200 New Jersey Avenue SE
Washington, D.C. 20590**

**IAEA CERTIFICATE OF COMPETENT AUTHORITY
SPECIAL FORM RADIOACTIVE MATERIALS
CERTIFICATE USA/0632/S-96, REVISION 5**

This certifies that the sources described have been demonstrated to meet the regulatory requirements for special form radioactive material as prescribed in the regulations of the International Atomic Energy Agency[1] and the United States of America[2] for the transport of radioactive material.

1. Source Identification - QSA Global, Inc. Model Nos. AX1, X.1, and X.1/2 (All models manufactured on or after May 17, 1977).

2. Source Description - Cylindrical double encapsulations made of stainless steel and tungsten inert gas or laser seal welded. Approximate outer dimensions of all models are 7.9 mm (0.31 in.) in diameter and 10.15 mm (0.4 in.) in length. Construction shall be in accordance with attached AEA Technology QSA, Inc. Drawing No. RBA10880, Rev. E.

3. Radioactive Contents - No more than either 3.7 GBq (100.0 mCi) of Americium-241 or 13.0 GBq (351.0 mCi) of Californium-252. The Am-241 is in oxide form and mixed with beryllium powder and pressed into a solid pellet. The Cf-252 is in the form of a metal wire or an oxide solid ceramic.

4. Quality Assurance - Records of Quality Assurance activities required by Paragraph 310 of the IAEA regulations[1] shall be maintained and made available to the authorized officials for at least three years after the last shipment authorized by this certificate. Consignors in the United States exporting shipments under this certificate shall satisfy the applicable requirements of Subpart H of 10 CFR 71.

5. Expiration Date - This certificate expires on March 31, 2013.

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[1] "Regulations for the Safe Transport of Radioactive Material, 1996 Edition (Revised), No. TS-R-1 (ST-1, Revised)," published by the International Atomic Energy Agency(IAEA), Vienna, Austria.

[2] Title 49, Code of Federal Regulations, Parts 100-199, United States of America.

(- 2 -)

CERTIFICATE USA/0632/S-96, REVISION 5

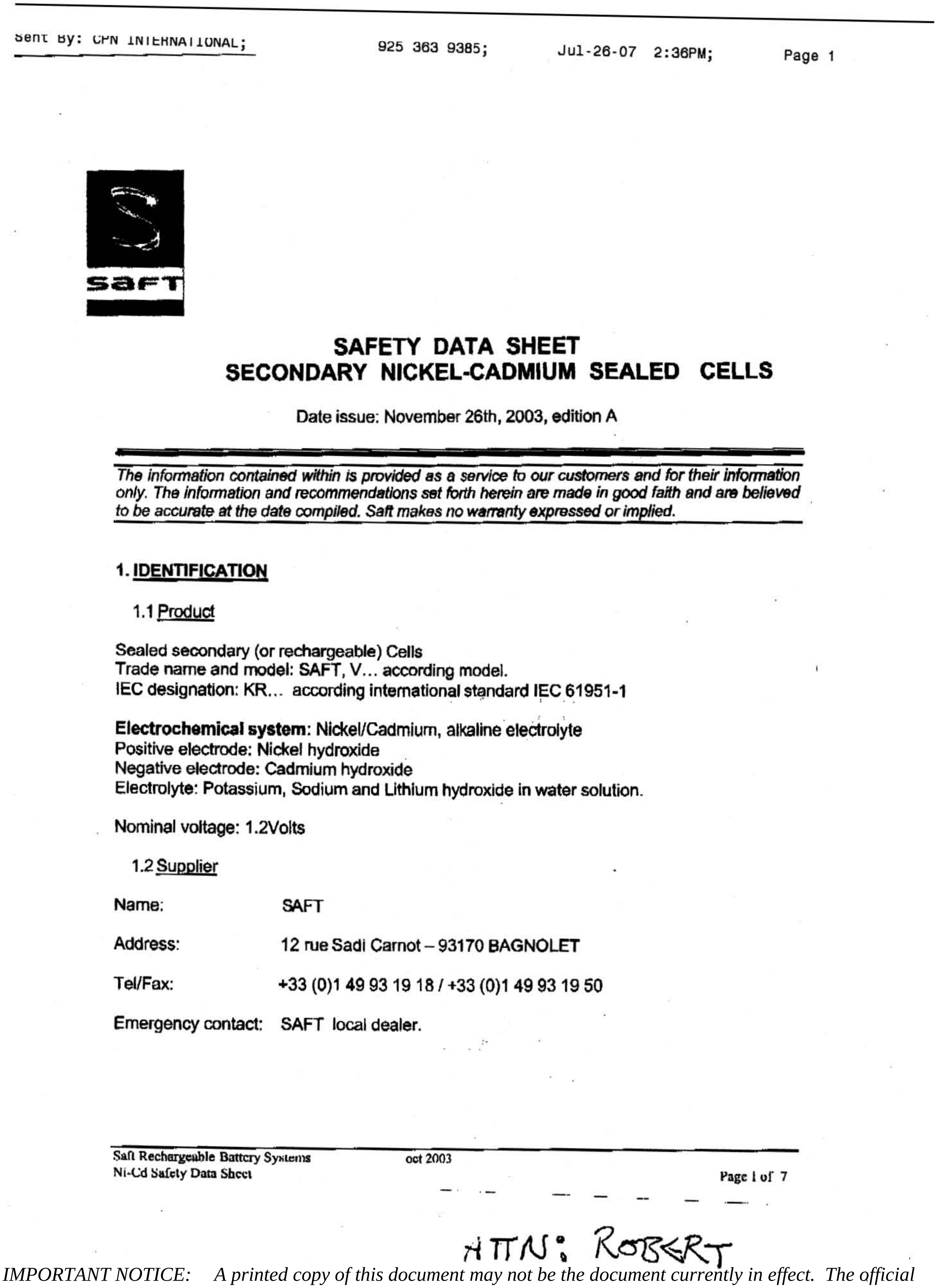
This certificate is issued in accordance with paragraph 804 of the IAEA Regulations and Section 173.476 of Title 49 of the Code of Federal Regulations, in response to the February 29, 2008 petition by QSA Global, Inc., Burlington, MA, and in consideration of other information on file in this Office.

Certified By:

Mar 13 2008
(DATE)

for Robert A. Richard
Deputy Associate Administrator for Hazardous Materials Safety

Revision 5 - Issued to extend the expiration date.



Sent By: CPN INTERNATIONAL; 925 363 9385; Jul-26-07 2:36PM; Page 1

saft

SAFETY DATA SHEET
SECONDARY NICKEL-CADMIUM SEALED CELLS

Date issue: November 26th, 2003, edition A

The information contained within is provided as a service to our customers and for their information only. The information and recommendations set forth herein are made in good faith and are believed to be accurate at the date compiled. Saft makes no warranty expressed or implied.

1. IDENTIFICATION

1.1 Product

Sealed secondary (or rechargeable) Cells
Trade name and model: SAFT, V… according model.
IEC designation: KR… according international standard IEC 61951-1

Electrochemical system: Nickel/Cadmium, alkaline electrolyte
Positive electrode: Nickel hydroxide
Negative electrode: Cadmium hydroxide
Electrolyte: Potassium, Sodium and Lithium hydroxide in water solution.

Nominal voltage: 1.2Volts

1.2 Supplier

Name:	SAFT
Address:	12 rue Sadi Carnot – 93170 BAGNOLET
Tel/Fax:	+33 (0)1 49 93 19 18 / +33 (0)1 49 93 19 50
Emergency contact:	SAFT local dealer.

ATTN: ROBERT

2. COMPOSITION (Weight percentage of basic materials)

Single cell with steel container

Metals		%	Plastics		%	Other		%
Iron	Fe	25 - 37	Polyamide	PA/PP	2.5 - 3.5	Potassium	K/Na/Li	1.8 - 2.9
Nickel	Ni	20 - 28	Rubber	EPDM	< 0.05	Water	H2O	4 – 9
Cadmium	Cd	10 - 15	Polyethylene	PE	0.2 - 0.4	OH-		8 -14
Cobalt	Co	0.4 -1.0	PVC		0.2 - 0.7			

3. HAZARDS

A- Human hazards

A sealed Nickel-Cadmium cell is not hazardous in normal use.

3.1 Physical

Nickel plated steel can do not present any risk if cells are used for its intended purpose and according to valid directions for use.

3.2 Chemical

Nickel plated steel can do not present chemical risk in normal use.

In case of misuse (abusive over charge, reverse charge, external short circuit...) and in case of default, some electrolyte can leak from the cell through the safety vent.
In these cases refer to the risk of the Alcaline hydroxides.

The toxic properties of the electrode materials are hazardous only if the materials are released by mechanical damaging the cell or if exposed to fire.

Classification of dangerous substances contained into the cells.

SUBSTANCES			CLASSIFICATION			
Name	EEC Number CAS Number	Symbol	Letter	Identification of danger	Special risk (1)	Safety advice (2)
Cadmium Hydroxide	048-001-00-5 21041-95-2	Cd(OH)2	Xn	Harmful	R 20/21/22	S 22
Nickel Hydroxide	028-008-x* 12054-48-7	Ni(OH)2	Xn	Harmful	R 20/22-43-40	S 22/36
Cobalt Hydroxide	- 21041-93-0	Co(OH)2	Xn	Harmful	R22-42/43	S22-24-37
Alcalines hydroxide	019-002-00-8 1310-58-3	KOH NaOH LiOH	C	Corrosive	R 35	S 26-37/39 -45

(1) Nature of special risk

R 20/21/22 :	Harmful by inhalation, skin contact or if swallowed.
R 20/22:	Harmful by inhalation or if swallowed.
R 35:	Causes serious burns.
R 40:	Possible risk of irreversible effects.
R 43:	May cause sensitising by skin contact.
R42/43:	May cause sensitising by inhalation and skin contact.

(2) Safety advice

S 22:	Do not breathe dust.
S 24:	Avoid contact with skin
S 26:	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S 36:	Wear suitable protection clothing.
S 37:	Wear suitable gloves.
S 37/39:	Wear suitable gloves and eyes/face protection.
S 45:	In case of accident or if you feel unwell, seek medical advice immediately.

B- Ecological hazards

Metals used in a Ni-Cd cell, and specifically the cadmium, have to be collected and recycled.

4. FIRST AID MEASURES

In case of electrolyte solution spill (cell leakage) precautions must be taken to avoid any contact of human tissues. If it accidentally happens following must be done:

4.1 Inhalation
Fresh air. Rinse mouth and nose with water. Medical treatment.

4.2 Skin contact
Rinse immediately with plenty of water. Medical treatment.

4.3 Eyes contact
Rinse immediately with plenty of water during at least 15-30 min .Immediate hospital treatment. Consult eye specialist.

4.4 Ingestion
If the injured is fully conscious: plenty of drink, preferably milk. Do not induce vomiting. Immediate Hospital treatment.

From: Schwers, Norman F
Sent: Thursday, October 28, 2010 3:38 PM
To: Ziock, Robert
Cc: Colborg, Shawn P; Spangler, Richie
Subject: FW: De Minimis quantity

Robert,

Based on our conversation, you would like to ship this same item /material to the manufacturer for calibration.
Based on the material type and quantity remaining the same as the previous analysis, the material remains below the DeMinimis requirements.
No NCS controls are required for this material.
I have included Shawn and Richie on this email since I assume that it will pass through 957.
Please pass this along to any of the shipping and packaging personnel or transportation personnel that may need this review.
If you have any questions about this review, please contact me.

Norm Schwers
NCSE
nfschwe@sandia.gov
845-3346

From: Schwers, Norman F
Sent: Tuesday, January 27, 2009 10:39 AM
To: Ziock, Robert; Archibeque, Edward A; Garcia, Andy J; Kidd, Carter R; Gabaldon, Glen G
Subject: De Minimis quantity

Robert,

I just received you call for the Am-241 shipment.
According to your call, the material is 1.85GBq (1.85E9 Bq) and the SNL De Minimus limit is 1.27E12 Bq.
This is less then the de Minimis quantity and can be stored in 957c with no Nuclear Criticality Safety controls required.
I am not sure who you will be working with in transportation or at 957c, so I included a few of the personnel that I know.
If you have any questions, just let me know.
Thanks
Norm Schwers
845-3346

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APPENDIX G
Mixed Waste Landfill Neutron Count Log Form

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Mixed Waste Landfill Neutron Logging Data Field Form (page 1 of 2)

Date:		Standard Count:
Start Time:		Chi:
Personnel:		Previous Count:
		Count Time: 30 seconds

Vertical Depth Below Top of Casing (ft)	Linear Depth Along Casing (ft)	Winch Counter Reading (ft)	VZ-3 Counts (E Side)	VZ-2 Counts (SW Corner)	VZ-1 Counts (NW Corner)
0.0	0	0			
0.9	1	9999			
1.7	2	9998			
2.6	3	9997			
3.5	4	9996			
4.3	5	9995			
5.2	6	9994			
6.1	7	9993			
6.9	8	9992			
7.8	9	9991			
8.7	10	9990			
9.5	11	9989			
10.4	12	9988			
11.3	13	9987			
12.1	14	9986			
13.0	15	9985			
13.9	16	9984			
14.7	17	9983			
15.6	18	9982			
16.5	19	9981			
17.3	20	9980			
18.2	21	9979			
19.1	22	9978			
19.9	23	9977			
20.8	24	9976			
21.7	25	9975			

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Mixed Waste Landfill Neutron Logging Data Field Form (page 2 of 2)

Vertical Depth Below Top of Casing (ft)	Linear Depth Along Casing (ft)	Winch Counter Reading (ft)	VZ-3 Counts (E Side)	VZ-2 Counts (SW Corner)	VZ-1 Counts (NW Corner)
26.0	30	9970			
30.3	35	9965			
34.6	40	9960			
39.0	45	9955			
43.3	50	9950			
47.6	55	9945			
52.0	60	9940			
56.3	65	9935			
60.6	70	9930			
65.0	75	9925			
69.3	80	9920			
73.6	85	9915			
77.9	90	9910			
82.3	95	9905			
86.6	100	9900			
90.9	105	9895			
95.3	110	9890			
99.6	115	9885			
103.9	120	9880			
108.3	125	9875			
112.6	130	9870			
116.9	135	9865			
121.2	140	9860			
125.6	145	9855			
129.9	150	9850			
134.2	155	9845			
138.6	160	9840			
142.9	165	9835			
147.2	170	9830			
151.6	175	9825			
155.9	180	9820			
160.2	185	9815			
164.5	190	9810			
168.9	195	9805			
173.2	200	9800			

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