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NMED Hazardous Waste Bureau

Mr. John E. Kieling
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Subject: Submittal of Updated Reference Document Cited in the Chemical Waste Landfill Post Closure Care Permit for the Department of Energy National Nuclear Security Administration/Sandia Site Office and Sandia National Laboratories/New Mexico, Environmental Protection Agency Identification Number NM5890110518

Dear Mr. Kieling:

The Department of Energy National Nuclear Security Administration/Sandia Site Office and Sandia Corporation are submitting the enclosed reference document to the New Mexico Environment Department in accordance with the requirements of the Chemical Waste Landfill Post-Closure Care Permit, Permit Attachment 2, Section 2.0 and Permit Attachment 3, Section 3.9. This Reference Document, *Sandia National Laboratories/New Mexico Statement of Work for Analytical Laboratories*, is listed in:

- Permit Attachment 2, Table 2-1; and
- Permit Attachment 3, Table 3-2.

Revisions include updates to keep the Reference Document current and to reflect ongoing modifications and improvements in industry practices. The revised Reference Document became effective on January 2, 2013.

If you have questions please contact me at (505) 845-6036 or John Weckerle of my staff at (505) 845-6026.

Sincerely,

Geoffrey L. Beausoleil
Manager

Enclosure

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Submittal of Updated Procedure
Chemical Waste Landfill Post-Closure Care Permit

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.

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Sandia Corporation
Albuquerque, New Mexico
Operator

1/17/13
Date signed

Geoffrey L. Beausoleil, Manager
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National Nuclear Security Administration
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Owner

24 JAN 2013
Date signed

Enclosure

Sandia National Laboratories

Statement of Work for Analytical Laboratories
Revision 6
January 2, 2013

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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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.

[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 <u>no less than 3:1</u> 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.

$$MDA = \frac{4.65S_g + 2.71}{2.22DEIVTRA}$$

$$L_c = \frac{2.33S_g}{2.22DEIVTRA}$$

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:

$$CU(cpm) = \sqrt{\frac{CR_{smp}}{T_{smp}} + \frac{CR_{bkg}}{T_{bkg}}}$$

The general form of the sample 1σ Total Propagated Uncertainty (TPU) equation is:

$$\sigma Conc\left(\frac{pCi}{g,L}\right) = |Conc| \times \sqrt{\left(\frac{CU}{CR_{smp} - CR_{bkg}}\right)^2 + \left(\frac{\sigma Eff}{Eff}\right)^2 + \left\langle \left(\frac{\sigma Decay}{Decay}\right)^2 - \text{or} - \left(\frac{\sigma Ingrowth}{Ingrowth}\right)^2 \right\rangle + \left(\frac{\sigma branch}{Branch}\right)^2 + \left(\frac{\sigma Yield}{Yield}\right)^2 + \left(\frac{\sigma aliquot}{Aliquot}\right)^2}$$

where:

$$Decay = \exp\left[\frac{-\ln 2 \times \Delta T}{HL}\right]$$

$$\sigma_{Decay} = \frac{Decay \times \ln 2 \times \sigma_{HL} \times \Delta T}{HL^2}$$

$$Ingrowth = 1 - \exp\left[\frac{-\ln 2 \times \Delta T}{HL}\right]$$

$$\sigma_{Ingrowth} = \frac{(1 - Ingrowth) \times \ln 2 \times \sigma_{HL} \times \Delta T}{HL^2}$$

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 $\pm$ 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 $\pm$ 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 $\pm$ 20 percent (approximately 3σ). Above the PQL, the individual sample isotope ratio control limits should be near the population mean $\pm$ 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:

$$pCi\ ^{235}U = (ug\ ^{235}U)/22.0$$

$$pCi\ ^{238}U = (ug\ ^{238}U)/3.33$$

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 $\geq$ 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) Method blank (preparation blank) analysis

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) Instrument blank analysis

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) Storage blank analysis

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. Laboratories shall not use silica sand or any other matrix substitute in PBs for solid sample analyses. 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) Solid LCS materials shall be analyzed with each batch of solid samples when such materials are available. 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) Alpha spectrometry

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 <u>only</u> 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

Analyte	MDL (Method 6010/6020) 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.

Radionuclide	MDA				
	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

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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

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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		

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		

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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	MDLs 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

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	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

Compound Name	CAS No.	MDLs	
		Water µg/L	Solid 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		

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Organic Parameters and Target MDLs

Table VI. (concluded) 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
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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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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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

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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

Attachment 3
Organic Parameters and Target MDLs

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	Holding Times Extract
2310B,2320B	Acidity, Alkalinity	Water	500 mL Plastic or Glass	≤6 °C	14 Days	NA
300.0, 300.1 375.2	Bromide, Chloride, Fluoride, Sulfate	Water	1 L Plastic	≤6 °C	28 Days	NA
5210B	BOD	Water	1 L Plastic	≤6 °C	48 Hours	NA
9010B, 9013, 9014, , 335.4, 4500CN-G	Total Cyanide Amenable Cyanide	Water Solid/Other	1 L Plastic 125 mL Glass Jar	≤6°C; NaOH; pH > 12 ≤6°C	14 Days 14 Days	NA NA
5310B, C or D, 9060	DOC, TOC	Water	250 mL Amber Glass	≤6 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	≤6 °C	28 Days	NA
200.7, 200.8, 6010B, 6020	All metals except Cr(VI) and Hg	Water Solid/Other	500 mL Plastic 250 mL Glass Jar	HNO_3; pH < 2	180 Days 180 Days	NA NA
3060A 218.6 7197, 7196A	Cr(VI)	Water Water Solid/Other	500 mL Plastic 500 mL Plastic 250 mL Glass Jar	≤6 °C ≤6°C, pH 9-9.5 ≤6 °C	24 Hours 28 Days 30 Days	NA NA 7 Days
245.1, 7470A, 7471A	Hg	Water Solid/Other	500 mL Plastic 250 mL Glass Jar	HNO_3; pH < 2 ≤6 °C	28 Days 28 Days	NA NA
130.1	Hardness	Water		HNO_3; pH < 2 ≤6 °C	180 Days	NA
345.1	Iodide	Water	500 mL Plastic or Glass	≤6 °C	24 Hours	NA
353.2, 351.1 351.2, 365.4 350.1	Ammonium, Nitrate + Nitrite, Total Phosphorus, TKN	Water	1 L Plastic	≤6 °C; H_2SO_4; pH < 2 ≤6°C; not acidified	28 Days 24 Hours	NA NA
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 Solid/Other	1 L Plastic 250 mL Glass Jar	≤6 °C; 1M Boric Acid ≤6 °C	48 Hours 48 Hours	NA NA

Attachment 5
Holding Times and Preservation Techniques

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times Extract
314.0, 9058	Perchlorate by IC	Water	250 mL Plastic or Glass	≤6 °C	28 Days	NA
6850 (modified) 6860 (modified)	Perchlorate by LC/MS/MS	Water	250 mL Plastic or Glass	≤6 °C	28 Days	60 days
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

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times 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 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	≤6 °C ≤6 °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	≤6 °C; HCl; pH < 2 ≤6 °C	14 Days 14 Days	NA 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 Soil/Other	3 x 40 mL Glass Vial 125 mL Glass Jar	≤6 °C; HCl; pH < 2 ≤6°C	14 Days 14 Days	NA 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 Soil/Other	4 L Amber Glass Bottle 250 Glass Jar	≤6 °C ≤6 °C	7 Days 14 Days	40 Days 40 Days
8082	PCBs	Water Soil/Other	4 L Amber Glass Bottle 250 Glass Jar	≤6 °C ≤6 °C	1 Year 1 Year	1 Year 1 Year
8141A	Organophosphorous Compounds	Water Soil/Other	4 L Amber Glass Bottle 250 Glass Jar	≤6 °C; NaOH or H_2SO_4; pH 5-8 ≤6 °C	7 Days 14 Days	40 Days 40 Days

Attachment 5
Holding Times and Preservation Techniques

Method	Parameters	Matrix	Volume/Container	Preservation	Holding Times Sample	Holding Times 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 Type	Container Vol	Preser-vative	Collect Method	Sample Type	Parameter & Method Requested	Lab Sample Id

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.

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.

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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)

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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)

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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.

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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 <u>only</u> 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