

# **Model Statement of Work For Analytical Laboratories**

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## STATEMENT OF WORK

### DOE-AL FACILITY GENERAL INORGANIC, ORGANIC, RADIOCHEMICAL, ASBESTOS, AND GEOTECHNICAL LABORATORY ANALYSES

#### Introduction

The U.S. Department of Energy Albuquerque Operations Office (DOE-AL) facility sample management office (SMO) is responsible for acquiring analytical services in support of facility activities. This statement of work (SOW) outlines the requirements for analytical services provided to the DOE-AL facility by laboratories.

Samples obtained for chemical analysis in support of DOE-AL facility activities will consist of soil, waste, groundwater, surface water, domestic supply water, air filters, demolition debris, biota, sludge, organic liquids, swipes, gas canisters, and bioassay samples. In addition, samples may be acquired for airborne asbestos, bulk asbestos, or geotechnical testing. The sections below detail specific quality assurance (QA) protocols, analytical practices and procedures, analytical quality control (QC) requirements, deliverable formats, and schedule requirements. Collectively, these conventions have been established to ensure that DOE-AL facility data quality objectives (DQO) are met and that data obtained from different laboratories are comparable. **(Discuss anticipated levels of radioactivity in this paragraph.)**

Laboratories may submit proposals for general inorganic and organic analysis, radiochemical analysis, asbestos analysis, geotechnical analysis, or any combination of these four major categories of services. Proposals for general inorganic analysis only, or organic analysis only, will not be accepted.

Laboratories shall perform all analyses of samples received. Laboratories shall obtain express written permission before sending any samples to a secondary laboratory for analysis. Secondary laboratories are generally required to successfully pass a DOE-AL systems audit and submit analysis results for performance evaluation samples prior to providing analytical support. In the event that a secondary laboratory is approved and does receive samples, all delivery schedules shall remain unchanged.

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GLOSSARY

## 1.0 ANALYSIS REQUESTS AND SAMPLE SHIPMENT

### 1.1 Work orders

Approximately one week before the start of sample collection, the DOE-AL facility contract representative will notify the contractor, in writing, of the scheduled shipment of samples. The notification shall take the form of a delivery order issued against the contract. The delivery order will include analysis request forms, which detail the samples to be submitted and the analyses requested. Some small projects and/or rapid analysis requests may be submitted without the advance notification discussed above in order to meet project requirements. **(Insert the correct details for your program here.)**

### 1.2 Sample custody

Sample custody is transferred to the laboratory at the time of sample receipt, after which the laboratory is responsible for maintenance of unbroken chain of custody (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. Section 2.9 of this document provides additional detail regarding sample receipt procedures.

### 1.3 Shipping charges

Sample shipping charges shall typically be paid by the DOE-AL facility. However, it may become necessary on occasion to ship samples collect. In such cases, the contractor shall be reimbursed for the shipping charges. Collect-shipping charges shall be included in the invoice for the associated delivery order.

### 1.4 Itemized analytical charges

Unit prices provided by laboratories shall include the cost of all quality assurance, quality control, preparation, extraction, cleanup, analytical, reporting, storage, and disposal requirements specified in this SOW. Field quality control 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.

### 1.5 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.

### 1.6 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.

### 1.7 Request for reanalysis

### 1.7.1 Incomplete reports and errors

In the event that 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 DOE-AL facility SMO reserves the right to request delivery of the missing documentation or the reanalysis of any or all samples within the sample lot. Reanalysis requests will be for the affected parameters only, rather than the entire analytical suite, where some results in the suite have met the acceptance criteria.

### 1.7.2 Reimbursement for reanalysis costs

Reanalyses will be requested by telephone and confirmed in writing. Payment for reanalyses requested by the DOE-AL facility SMO shall be made according to the following guidelines:

- a) Reanalyses requested because failed QC data were reported to the DOE-AL facility SMO shall not be paid for.
- b) Reanalyses that are requested because of a suspected significant error, and that confirm the original results within reasonable analytical error, shall be paid for by the DOE-AL facility. The DOE-AL facility SMO 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 the DOE-AL facility.

## 1.8 Non-standard analyses

### 1.8.1 Bids for non-standard analyses

The DOE-AL facility 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 DOE-AL facility SMO 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.

### 1.8.2 Proposing alternate laboratories

If none of the laboratories holding current contracts have the capability to perform the needed test, laboratories will be allowed to propose alternate laboratories outside the current contract structure. If one or more of the proposals is accepted, the laboratory shall be solely responsible for executing a contract with the proposed laboratory for the work. An assessment of the need to perform an audit of the proposed laboratory prior to submitting samples will be made by the

DOE-AL facility SMO. However, the laboratory 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.

### 1.8.3 Using alternate laboratories

Laboratories shall not be permitted to send DOE-AL facility samples to laboratories outside the original contract structure unless the conditions described in sections 1.8.1 and 1.8.2 have been met.

## 2.0 QUALITY ASSURANCE REQUIREMENTS

### 2.1 General data quality objectives

#### 2.1.1 Methods, quality control, and documentation

- a) The DOE-AL facility will sometimes find, through application of the DOE “SAFER” process for DQOs, that the quality control 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.
- b) As discussed above, DQOs are developed by the DOE-AL facility project specifically. However, a general requirement is that industry-standard methods, such as USEPA SW-846 (Third Edition), 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. When necessary, the analytical requests submitted to a laboratory will specify which methods apply. In the absence of specific direction from the facility, laboratories may elect to use any industry-standard set of methods. Where industry-standard methods do not address particular analytes, performance-based methods may be utilized with prior approval from the DOE-AL facility SMO. **(Insert specific site requirements here.)**
- 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 and the U.S. Environmental Protection Agency (EPA) (or other industry-standard) methods cannot be extended to requested parameters, professional judgment shall be employed in adhering as closely as possible to the spirit of those protocols. This means that the laboratory should extend all standard documentation and quality control practices to

parameters, methods, and analytical techniques that are not covered in SW-846 or this SOW 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 DOE-AL 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 Deliverable levels

DOE-AL facility data deliverables routinely provided by the laboratory will be "Level D" reports. Laboratories will provide "Level C" reports upon request, and will provide line item pricing for each type of report in the schedule of charges submitted to the DOE-AL facility. Reporting level definitions and analytical data deliverable requirements are fully outlined in section 4 of this document. **(Modify to specify the correct routine deliverable for your facility.)**

#### 2.1.3 NELAP Certification

Unless specifically allowed exemption by the DOE-AL facility SMO, laboratories serving DOE-AL facilities must be certified by the NELAP national accrediting organization and the State of Utah.

### 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) which contains sections 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) Instrument calibration procedures, including documentation of calibration standards, coefficients resulting from linear or higher order polynomial regression calculations, and calibration curve correlation coefficients. 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 for general inorganic chemistry be  $\geq 0.995$ .
  - 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 of 0.99. If higher order polynomials are used, laboratories must obtain prior approval from the DOE-AL facility SMO and must follow the applicable guidance in SW-846 method 8000B.
  - iii. Calibration frequency, methodologies, and documentation practices for radiochemistry counting instruments shall be discussed.
- i) Method detection limits (MDL) for general inorganic chemistry and organic chemistry. The section addressing MDLs shall specify detection limit determination methodologies. Minimum MDL study requirements are discussed in greater detail in section 3.3.1 of this SOW. Minimum detectable concentration (MDC) calculation requirements for radiochemistry are given in section 3.3.4 of this SOW.
- j) The LQAP shall specify default criteria for QC sample type, analysis frequency, data acceptance, and corrective actions for failures in daily QC practices. 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 DOE-AL facility work are discussed in detail in section 3 of this SOW.
- k) The corrective action report (CAR) process shall be described and a copy of a CAR form shall be provided in the LQAP.
- l) The laboratory document-control procedures shall be described. In addition, the LQAP shall outline document flow, including review steps, from COC to the final analytical report.
- m) The process for data review and approval shall be outlined in the LQAP. Provision shall be made for peer, supervisory, or QA review of all chemist worksheets.
- n) The laboratory's holding time policies and processes for pre-preservation of sample bottles, sample preservation checks, and documentation of preservation checks shall be discussed in the LQAP. Holding times and

preservation techniques for DOE-AL facility samples are outlined in Attachment 5.

- o) The frequency and method of conducting and documenting internal audits shall be discussed. In addition, the LQAP shall specify the frequency and contents of QA reports to management.
- p) The LQAP shall list approvals and certifications from states and external agencies.
- q) The LQAP shall specify the laboratory policy regarding the number of significant figures to be used in reporting analytical results. Also, the LQAP or an SOP shall require the use of leading zeros 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 4.1.12.) Additionally, the LQAP or an SOP shall define appropriate error correction practices and require the use of indelible ink for records.
- r) The LQAP shall describe procedures for material procurement, quality inspection, inventory, and storage.
- s) The LQAP shall discuss methods for verification of electronic data deliverable (EDD) and hard copy agreement for sample identifiers, results, detection limits, uncertainties, and QC data.

#### 2.2.2 SOP support for the LQAP

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

Chemical analysis laboratories shall perform the analysis of performance evaluation (PE) samples provided to the laboratory by the DOE-AL facility (**insert schedule here**) if requested to do so. The analytical and deliverable requirements for these PE samples are the same as all DOE-AL facility samples. Payment for the analysis of PE samples shall be made according to the fees specified in the contract. The DOE-AL facility will not pay for the analysis of "known" samples provided as a courtesy for quality control, investigations, or method development.

#### 2.3.2 PE 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 DOE-AL facility samples.

### 2.3.3 Proficiency

- a) A summary of analytical results and theoretical values for each PE round will be provided to each laboratory by the DOE-AL facility SMO after all the data for that round are in. 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 DOE-AL facility SMO 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 DOE-AL facility chemical analysis program.
- b) Laboratory performance information may be shared among DOE-AL facilities and entities supporting DOE-AL site activities. The DOE-AL Analytical Management Program policy governing the sharing of contractor performance information is provided as **Attachment XX. (Number the Attachment.)**

## 2.4 Systems and internal audit requirements

### 2.4.1 Annual systems audits

The laboratory shall undergo a DOE-AL facility systems audit at least once per year. The purpose of this audit 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 audit report will be issued following this activity. Responses to audit reports will be due 30 days from the date of issue.

### 2.4.2 On-site data package review

Data package reviews may be conducted at the laboratory at the discretion of the DOE-AL facility. 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 will be issued following this activity. 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. The results of the laboratory's internal QA audits shall be provided to the DOE-AL facility SMO in the next quarterly progress report (QPR). QPRs are discussed in section 2.14.

## 2.5 Participation in inter-laboratory comparison studies

### 2.5.1 Required intercomparison programs

The laboratory shall participate, where appropriate, in the inter-laboratory comparison studies administered by the agencies listed below. Laboratories performing chemical analyses of DOE-AL facility samples under an organic and general inorganic contract shall participate in (a) and (c), below. Laboratories performing chemical analyses under a radiochemical contract shall participate in (b) and (c), below. Laboratories performing airborne silica, asbestos, metals, and/or organics analyses shall participate in (d), below. Laboratories performing lead in paint analyses shall participate in (e), below. Laboratories performing NPDES analyses shall participate in (f), below.

- a) Commercial vendor program designed to meet the requirements given in the Proficiency Testing section (Chapter II) of the NELAC standard.
- b) Inter-laboratory Quality Assurance Program (QAP), U.S. Department of Energy, Environmental Measurements Laboratory (EML), New York, New York.
- c) Mixed Analyte Performance Evaluation Program (MAPEP), U.S. Department of Energy, Idaho Operations Office, Idaho Falls, Idaho.
- d) Proficiency Analytical Testing Program (PAT), American Industrial Hygiene Association (AIHA).
- e) Environmental Lead Proficiency Analytical Testing Program (ELPAT), AIHA.
- f) Discharge Monitoring Report--Quality Assurance Study (DMR-QA), EPA Office of Enforcement and Compliance Assurance.
- g) **Add radon, bioassay, or other PE programs as required for your site.**

### 2.5.2 Reporting intercomparison results

The laboratory shall report results of the intercomparison studies specified in section 2.5.1 to the DOE-AL facility SMO quarterly. This report is due on the 15<sup>th</sup> day of January, April, July, and October to coincide with the delivery dates for QPRs. 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 DOE-AL facility laboratory analysis program.

### 2.6 Employee training and documenting employee proficiency

The DOE-AL facility 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. Personnel that have not been trained and evaluated shall not participate in the handling or analysis of DOE-AL facility samples.

Evidence files must exist to demonstrate that each employee has met the laboratory's minimum training requirements and has read, 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 their 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 DOE-AL facility 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 DOE-AL facility. 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 DOE-AL facility 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 DOE-AL facility 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 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 DOE-AL facility 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  $\pm$  one 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 range  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . 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  $\mu\text{L}$  or more. This SOP shall specify that pipettes be checked daily (on all business days) by weighing deionized (DI) water, and that pipettes failing to deliver to within  $\pm$  one 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 ten mg/L or higher. General inorganic analyte solutions with constituent concentrations less than ten 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.

Volatiles	7 days for gases 180 days for non gases
Total petroleum hydrocarbons	180 days for purgeable (GRO) 365 days for extractable (DRO)
Pesticides/PCBs/herbicides	180 days
Semi-volatiles	365 days
High Explosives	365 days, $\geq 1,000$ 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 axial viewing ICP-AES working standards 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 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, which ever 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. If verification is allowed, the methodology to be 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 manufacturer's 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 M $\Omega$ -cm resistivity control criterion. Preparation water need not meet the newer 18.0 M $\Omega$ -cm criterion.
- l) The SOP shall require that standards for radiochemistry and wet chemistry be prepared using ASTM Type II water, at 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 DOE-AL facility work shall be ASTM Class A glassware. Dilutions may also be accomplished by automation or 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, H&S, QA, or other laboratory groups.

### 2.8 Analytical and QA SOPs

#### 2.8.1 Control of SOPs

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 DOE-AL facility samples shall be transmitted to the DOE-AL facility SMO 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 SOPs

Controlled copies of SOPs shall be readily available to all personnel performing analytical work in support of the DOE-AL facility. This may be accomplished either by issuing a copy to each analyst, or by making a library of SOPs accessible to analysts.

#### 2.8.3 Analyst familiarity with SOPs

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 DOE-AL facility 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 COC forms

DOE-AL facility samples received by the laboratory will be accompanied by a COC form. **An example of the COC/analysis request form is provided as Attachment XX.**

- a) At the time of sample receipt, this form will have been partially completed by the sampling team, and should indicate the laboratory name, delivery order number, sample IDs, sample matrix, bottle volume, collection dates and times, date shipped, and method of shipment. **(Insert the specifics for your program here.)**
- b) Individual sample bottles are labeled with the sample ID, sampling date and time, preservation method, sampler's identity, and comments. **(Insert the specifics for your program here.)**
- c) The laboratory sample custodian receiving the samples shall verify that the information listed on the COC 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

COC form in indelible ink to acknowledge sample receipt and accept custody. The sample custodian shall note discrepancies between the samples listed on the COC and those actually received on the COC 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 and 4.1.2 of this SOW for reporting requirements.)

### 2.9.3 Documentation of anomalies

The laboratory sample custodian shall note on the COC 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], 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 range for samples requiring cooling for preservation is  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . 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 SMO 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 DOE-AL facility SMO immediately by telephone of any irregularities noted during the sample receiving process. In addition, the laboratory shall notify the DOE-AL facility 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 DOE-AL facility samples associated with a specific delivery order for a period of 90 days after issuing the analytical report for that delivery order. **(Insert the specific requirements for your program here.)**

#### 2.9.6 Sample disposal

The laboratory is solely responsible for lawful disposal of all DOE-AL facility samples after the 90-day sample storage requirement is fulfilled if the exception given in (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 DOE-AL facility.
- b) If samples are to be returned to the DOE-AL facility, the laboratory shall provide notification that includes an inventory of samples to the DOE-AL facility SMO at least two weeks prior to shipping.
- c) Laboratories shall not return extracts or digestates to the DOE-AL facility.

#### 2.9.7 Return of shipping coolers

Laboratories shall initiate the shipment of sample coolers and blue ice back to the DOE-AL facility SMO within five days of receipt. Sample coolers should be shipped to the address given below unless different instructions are provided by the DOE-AL facility SMO. **(Insert compensation arrangements for return shipment here.)**

**(Insert correct address here.)**

#### 2.9.8 Laboratory-supplied sample containers

The DOE-AL facility may wish to use laboratory-supplied sample containers for some projects. The schedule of prices provided in the laboratory's proposal shall include separate prices for pre-cleaned sample containers, having the appropriate preservative, for each analysis covered in the proposal. Container and preservative requirements are given in Attachment 5 of this SOW.

#### 2.10 Holding time requirements

##### 2.10.1 DOE-AL facility 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. That is, if the EPA-specified holding time is given in hours, then the analysis must be complete before the end of the last hour of the holding time when calculated from the sampling time. When the holding

time is given in days, the analysis must be complete before the end of the day on which the holding time would expire as calculated from the sampling day.

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.

#### 2.10.3 Matrix types

Where matrix-specific holding times are not given in Attachment 5, the specified holding times 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 DOE-AL facility SMO 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 DOE-AL facility SMO approval will not be paid for.

- a) The DOE-AL facility SMO will make every effort to notify the laboratory when samples having less than 72 hours of the holding time remaining are to be shipped.
- b) For samples having 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 turn around premium price. That is, when 48 to 72 hours remain, the three day turn around price applies, and when less than 48 hours remain, the one day turn around price applies. Any deliverable schedule that is at least as long as that covered by the premium price may be requested by the DOE-AL facility SMO.

#### 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 DOE-AL facility SMO. Laboratories that do cancel analyses without notifying the DOE-AL facility SMO may be suspended from the DOE-AL facility laboratory analysis program.

### 2.11 Laboratory data verification and review requirements

#### 2.11.1 Worksheet review

- a) All analyst worksheets describing analysis of DOE-AL facility 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 DOE-AL facility SMO 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 DOE-AL facility SMO or discarded, at the discretion of the DOE-AL facility 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 CARs

The laboratory may be required to provide a CAR for any out-of-control event associated with analytical services provided to the DOE-AL facility.

##### 2.13.2 Delivery of CARs

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 DOE-AL facility SMO reserves the right to request delivery of CAR responses in less than two weeks if circumstances indicate that this is necessary. Failure to submit requested CARs may result in suspension of the laboratory from the DOE-AL facility laboratory analysis

program.

## 2.14 QPR requirement

### 2.14.1 Contents of QPRs

The laboratory shall submit QPRs to the DOE-AL facility SMO. QPRs shall address calendar quarters and are due by the 15<sup>th</sup> day of the month following the reporting period. In addition to the quarterly reporting requirement, laboratories will notify the DOE-AL facility SMO immediately for issues relating to items (d), (e), and (f). Emphasis should be placed on the following for inclusion in QPRs:

- a) New analysis methods and changes in old methods.
- b) Summaries of out-of-control incidents during the reporting period, and copies of the associated CARs.
- c) Descriptions of changes in the LQAP that affect the analysis of or documentation for DOE-AL facility samples.
- d) Changes in QA and key technical personnel, including resumes of new personnel. *Immediate notification of the applicable DOE-AL SMO personnel is also required in this case.*
- e) Changes in certification status with any regulatory or certifying agencies. *Immediate notification of the applicable DOE-AL SMO personnel is also required in this case.*
- f) Loss of capability to perform any service that is specified in existing contracts with DOE-AL facilities. *Immediate notification of the applicable DOE-AL SMO personnel is also required in this case.*
- g) Copies of performance evaluation feedback reports received during the reporting period.
- h) MDLs and QC limits if they are updated during the reporting period.

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

Failure to comply with the QPR requirement in this SOW may result in suspension of the laboratory from the DOE-AL facility laboratory analysis program.

## 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 DOE-AL facility samples.

### 2.15.2 DOE-AL facility SMO contact persons

The technical representatives in the DOE-AL facility SMO shall be **(give SMO staff names and titles here)**.

### 2.15.3 Communication

Open communication between the DOE-AL facility and laboratories is crucial to developing a mutually satisfactory business relationship. Laboratory technical representatives are encouraged to seek guidance in advance of performing work when any questions arise and 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 DOE-AL facility SMO 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. GALPs 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 of applicability, 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 quality assurance staff members monitor computer activities.
- c) Ensure that personnel, resources, and facilities are adequate and

available.

- d) Receive reports of audits of LIMS and 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) Have adequate education, training, and 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.
- c) Are of sufficient number for timely and proper operation of the computer systems.

#### 2.17.3 Quality assurance

The laboratory shall designate QA staff to monitor computer functions and procedures. QA staff members shall:

- a) Audit the computer systems at intervals adequate to ensure the integrity of the electronic data and prepare audit reports. Reports shall include a description of 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.
- b) Determine that no deviations from approved SOPs were made without proper authorization and adequate documentation.
- c) 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.
- d) Establish non-conformance 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 verification of 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 SOPs 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) Documentation for the issues in section (a) above is maintained. Laboratory management shall ensure that all documentation is readily available in the facility where the software is used.

#### 2.17.6 Security

Laboratory management shall ensure that the security practices to ensure the integrity of electronic data:

- a) Ensure that calculation routines are secure from inadvertent changes.
- b) Make login password necessary 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) Of adequate design and capacity, and that a written description is maintained.
- b) Installed and operated in accordance with manufacturer's recommendations and, at installation, undergo acceptance testing.
- c) Adequately 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 Records retention

Laboratory management shall ensure that SOPs for records retention are implemented and that the SOP specifications are followed by staff.

#### 2.17.9 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 adequate storage capacity is provided for retention of electronic data, storage media, and records pertaining to the computer systems.

#### 2.17.10 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 at a frequency adequate to ensure that they accurately describe the current procedures.
- c) SOPs are approved and changed in accordance with QA policy.
- d) A historical file of SOPs is maintained.

#### 2.18 Records for method development and initial demonstration of proficiency

The laboratory QAP 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

#### 3.1 Standard preparation and instrument calibration requirements

##### 3.1.1 Working standards

Standard preparation for analysis of DOE-AL facility samples shall be performed according to the specifications in (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. ICV solutions, if applicable, shall be documented in sufficient detail to make clear that they 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 (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. Laboratories analyzing As, Cd, Pb, Sb, Se, and Tl by axial-viewing ICP-AES rather than ICP-MS or GFAA shall, for each parameter, perform a four-point calibration of the instrument. Suggested standard concentrations are a blank, one standard in the 50-100 ppb range, one standard in the 250-500 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. When inorganic anions are analyzed by ion chromatography, 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  $\pm 10$  percent retention time control limits specified in method 9056 (section 7.1.4) and 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. Calibration for instruments used in organic chemistry is discussed in detail in section 3.5.2 of this SOW.

### 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. A completed analyst worksheet that includes the information listed below is required for each analytical run. Analyst worksheets may be computer generated or hand written using indelible ink.

- 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 SW-846 method unless different procedures are specified in the analysis request. Unless specifically asked to do otherwise by the DOE-AL facility 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) and solid phase extraction (SPE) are not allowed unless rigorous method development data are submitted and approved in advance.
- b) Laboratories working under general inorganic contracts shall specify digestion prices for SW-846 digestions, total dissolution digestions (HNO<sub>3</sub>, HClO<sub>4</sub>, HF), and toxicity characteristic leaching procedure (TCLP) extractions. All metals digestions and sample dilutions shall be performed using ASTM Type I water.

Unless specific exemption is given by the DOE-AL SMO, 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 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. This digestion requirement can also apply to uranium analysis by ICP-MS upon request by the DOE-AL facility SMO.
  - iv. The SMO will specify the sample preparation techniques for radionuclide determinations by general inorganic analytical techniques. This applies to any radionuclide determinations by column-extraction/flow-injection ICP-MS.
- d) Percent solids measurements shall be made and reported for all DOE-AL facility soil samples submitted for analyses other than tritium and geotechnical tests. Unless otherwise specified, soil sample results for all other analyses shall be reported on a dry weight basis.
  - e) Percent moisture data shall be reported for tritium analysis.
  - f) Extraction procedures for soil samples submitted for anion analysis will be selected on a case-by-case basis. The detection limits presented in the attachments assume a nominal 1:20 dilution factor.
  - g) **Add site requirements for digestion of swipes, filters, bioassay samples, and other matrices not covered in the requirements above.**

### 3.2.3 Initial dilution of samples

Since some DOE-AL facility samples have a high solids content, initial dilution of samples for analysis by GFAA, ICP-AES, and ICP-MS will be allowed according to the criterion specified in (a) below. Samples submitted for organic analyses may be initially diluted according to (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 integer dilution factor required to bring the solids content down to below 2000 mg/L. If TDS analysis was not requested by the DOE-AL facility SMO, a sample's specific conductance may be used to estimate TDS. For this purpose, the specific conductance of the unpreserved sample fraction in  $\mu\text{mho/cm}$ , 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.

- c) Samples diluted according to the criteria specified in (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 SOPs

The laboratory shall employ approved analytical techniques and SOPs in the analysis of DOE-AL facility samples. If a nonstandard technique is required to achieve a specific DOE-AL facility 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, National Institute for Occupational Health and Safety (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) For GFAA analyses, the laboratory shall adhere to the analytical and data qualification procedures specified in exhibit E, section V of EPA CLP SOW number ILMO4.0.
- c) If, due to catastrophic instrument failure, the specified technique(s) cannot be used, a laboratory representative shall contact the DOE-AL facility SMO 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 DOE-AL facility SMO.
- d) 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.
- e) When perchlorate is run by HPLC/MS/MS, daily calibration using linear regression shall be performed. Calibration shall consist of a blank and five standards. All  $\text{ClO}_4$  analyses will incorporate an appropriate internal standard to monitor ionization suppression and sensitivity fluctuations. The inorganic QC criteria for QC sample type, frequency, acceptance criteria, and corrective actions given in section 3.4 of this SOW shall be applied exactly unless they are specifically altered in the paragraphs below.

Second-source ICV shall immediately follow calibration, and a  $\pm 15$  percent acceptance criterion shall apply. That acceptance criterion shall also apply to subsequent CCVs. A detection limit verification standard shall be run in every batch. That standard will be called "CRI," will be prepared at approximately twice the MDL level, and must be recovered within  $\pm 30$  percent. If the  $\pm 30$  percent acceptance criterion is not met, the analyst must terminate the analysis, initiate corrective action, and successfully reanalyze the CRI before proceeding. Additionally, a low-concentration (approximately the MDL) standard addition for a sample slightly above the MDL is suggested but not required.

$\text{ClO}_4$  analyses performed by IC or LC/MS/MS will include use of the barium, silver, and hydrogen cleanup cartridges discussed in EPA method 314. Given the sensitivity of the technique under current conditions (low ppt detection limits), the use of all disposable labware in all cases is highly recommended to avoid cross contamination.

The transitions associated with both the  $^{37}\text{Cl}$  and  $^{35}\text{Cl}$  perchlorate ions (101 to 85 and 99 to 83 AMU transitions) shall be monitored in  $\text{ClO}_4$  analyses. The natural isotopic abundances for the chlorine isotopes gives a  $^{35}\text{Cl}/^{37}\text{Cl}$  ratio of approximately 3.1. Laboratories must statistically derive acceptance criteria for the isotope ratios obtained in field sample analyses to be used as an additional confirmation of analyte identity. Isotope ratio acceptance criteria should be derived using real sample matrices as opposed to a DI water matrix, or a combination of these (not DI alone). A variety of concentrations from the MDL level up to about 3 or 4 ppb should also be included in the isotope ratio study. Analytical results that subsequently fail the isotope ratio acceptance criteria should be flagged with an "X" qualifier, for which all documentation, reporting, and narrative requirements of this SOW shall apply.

Soil MDLs shall be determined separately using a prep that is identical to that used for field samples. Recommended soil preparation is as follows: Weigh 2 g of sample into a clean centrifuge tube. Add 20 mL 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, Ag, and H cartridges) and analysis.

### 3.3 Detection limits, reporting requirements, and QC exemptions

#### 3.3.1 MDL determination

The MDL for all organic and general inorganic parameters shall be determined, and verified (per section 3.3.1 (a) (iv)), and the results submitted to the DOE-AL facility SMO annually. Exceptions to this requirement are given in section (a) (vi) 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.

Unless superseded by new EPA guidance, MDL determinations shall be performed as specified in 40CFR136 and SW-846 Chapter I. 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. Empirical verification of MDLs shall be performed quarterly if the laboratory does not perform annual MDL studies. Empirical verifications of existing MDLs may be done with a single standard prepared at the MDL concentration.
  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 that 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.
  5. Guidance for documenting judgement 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 based upon the likelihood of producing false positive or false negative results. If false negative results are expected (see below), the MDL study should be repeated in any case. The DOE-AL facility SMO must approve the use of alternate values for MDLs.

We have encountered incorrect calculated MDLs many times in DOE-AL facility work, 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 that will 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 results in calculated MDLs that are too low. That is, laboratories cannot actually “see” 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 spirit of the 40 CFR 136 language, and to address the ubiquitous MDL problems we have encountered in DOE-AL facility 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.

- 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 DOE-AL facility SMO 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 DOE-AL facility target MDL, a discussion of the problem and planned corrective action shall accompany the report deliverable. (This requirement is waived where a prior agreement exists that 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. **(Insert your target MDLs in Attachments 1 and 3.)**

- c) The reports for MDLs should be in tabular summary form. 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 DOE-AL facility SMO 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 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.

If method blank “hits” exceed ten 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  $\sigma$  is the standard deviation of the positive method blank hits. The population of method blank results shall then be re-evaluated 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 DOE-AL Analytical Management Program staff if it fails to meet the objective of reducing false positives to single digit values.

### 3.3.2 Practical quantitation limits

Practical quantitation limits (PQL) shall be reported with all organic and general inorganic analysis results. PQLs are defined to be five times the MDL for the associated parameter. **DOE-AL facilities should insert or reference the facility-specific quantitation limit/reporting limit requirements here.**

### 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 sample specifically to reflect the conditions for that sample. That is, the MDLs and PQLs shall reflect dilution factors and sample aliquot sizes used in the analysis of each sample.
- c) Organic and general inorganic results that are less than the MDL shall be qualified with a “U” flag. Results between the MDL and the PQL shall be

qualified with a “J” flag.

### 3.3.4 Radiochemistry detection limits

**Use of the formulas and reporting conventions discussed below is strongly recommended. However, facilities may alter the requirements of section 3.3.4 as needed to accommodate site-specific needs.**

The DOE-AL facility 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 sections (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 (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 section (c). Also, should they wish to do so, laboratories may apply the detection limit calculation approach of section (c) to detectors having backgrounds above 35 counts.

- a) Radiochemistry laboratories shall calculate a sample-specific MDC for each radiochemical parameter. The MDC values shall be calculated according to the equation below and reported with each analytical result submitted to the DOE-AL facility 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 MDC, then the result of a subsequent count would have a five-percent probability of coming out below the decision level concentration (DLC). 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). **(Insert the target MDCs for your facility in Attachment 2.)**

$$\text{MDC} = \frac{4.65(\text{TBC})^{1/2} + 2.71}{2.22\text{DEIVTRA}}$$

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 DLC for each analytical result. A blank or sample will be considered to have activity above the applicable background only when the blank or sample concentration exceeds the DLC. DLC values shall be calculated according to the equation below and reported with each batch blank and sample result submitted to the DOE-AL facility SMO. This calculation gives the level at which there is a five-percent probability of reporting a false positive result for a sample containing no activity.

$$\text{DLC} = \frac{1.645(2 \cdot \text{TBC})^{1/2}}{2.22\text{DEIVTRA}}$$

Where, the variables are defined in the same way as those in the MDC calculation of section (a).

- c) Low background MDC and DLC

When at least 35 background counts cannot be obtained, laboratories shall use the equations given below to calculate MDC and DLC. 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 document, laboratories are not to use sand or any other matrix substitute in radiochemistry preparation blanks (PBs) associated with DOE-AL facility work. As a consequence, the digestion reagents and separation processes involved in the method define the blank type.

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

$$\text{DLC} = \frac{2.33S_g}{2.22\text{DEIVTRA}}$$

In these equations, the variables in the denominators are defined in the same way as those in the MDC calculation of section (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 MDC and DLC 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 MDC and DLC 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 MDC 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 MDC.”

### 3.3.5 Analytical uncertainty

- a) General inorganic and organic analytical results shall not be accompanied by estimates of uncertainty.
- b) 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, and the like, and calculate their contributions to uncertainty. All radiochemical result uncertainties shall incorporate terms for technique-related and sample-specific measurement errors.

### 3.3.6 QC 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 QC 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 quality control policies.

### 3.3.8 Batch QC requirements

The replicate and spike requirements given in this SOW apply to samples submitted by the DOE-AL facility. Laboratories shall not substitute replicate and spike data that were acquired for samples submitted by other clients. If multiple SDGs from one DOE-AL facility are batched together, then the relevant QC data must be included in each of the SDG reports.

### 3.3.9 Additional requirements for fluoride run by IC

Laboratories that run fluoride by ion chromatography 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.

## 3.4 General inorganic analytical QC requirements

Situations that make meeting the quality control 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 call the DOE-AL facility SMO to request an exemption from the reanalysis requirement for quality control 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 initial calibration verification (ICV) 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 two hours or after every tenth analytical sample, whichever is more frequent. The same standard shall be used for all 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 or PBs, spiked samples, laboratory control samples (LCSs), 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  $\pm$  ten percent of the known value except for cyanide ( $\pm$  15 percent) and mercury ( $\pm$  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. 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 the PQL, 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 any analyte concentration in the blank is in excess of the PQL, the lowest reported concentration in the associated samples must be at least ten times the concentration in the blank. All samples having that analyte's concentration less than ten times that of the blank but above the PQL shall be redigested and reanalyzed.

#### 3.4.4 Interference check samples (ICSs)

- 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 DOE-AL facility 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 analytes in the ICS (solution AB) shall agree within  $\pm 20$  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 DOE-AL facility SMO.

#### 3.4.5 Serial dilution

- a) One serial dilution analysis shall be performed in ICP-AES work for each matrix in every batch, with a minimum of one serial dilution analysis per 20 samples. 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 within  $\pm$  ten 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.

- b) One serial dilution analysis shall be performed in ICP-MS work for each matrix in every batch. 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 within  $\pm$  ten 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 X the reagent blank concentration, it should be much easier to implement in practice.

#### 3.4.6 Linear range verification

Quarterly linear range verification samples may be used to justify reporting ICP-AES or ICP-MS analytical results that exceed the calibration range. Results for the linear range verification sample must agree within  $\pm$  ten percent of the known value, and must be reported with the batch, if this approach is used. All other general inorganic (and organic) results reported must be within the calibration range.

#### 3.4.7 Laboratory control samples

LCSs shall be analyzed using the same sample preparation and analysis methods used for DOE-AL facility 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  $\pm$  20 percent of the true value for all general inorganic parameters.
- 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 developed by the laboratory. Under no circumstances shall a solid LCS be used when the applicable acceptance criteria exceed the 30-150 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.

- e) Exceptions to the reanalysis requirement are silver and antimony. LCS failures for silver and antimony shall be discussed in the case narrative but shall not be subject to the reanalysis requirement.

### 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, 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 programs may elect to allow or require MSD analyses to replace replicate analyses in all cases. **The DOE-AL facility SMO 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 five times the PQL. For samples with concentrations less than five times the PQL but greater than the PQL, the control limit is  $\pm$  PQL. No precision criterion applies to samples with concentrations less than the PQL.

- 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 4.1.9 (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.

The percent recovery for spiked samples is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{SSR}-\text{SR}}{\text{SA}} \times 100 \%$$

Where:

SSR = spiked sample result  
SR = sample result  
SA = spike added

- a) Matrix spikes shall be performed for all analytes other than sodium, potassium, magnesium, calcium, and the parameters listed in section 3.3.7. In addition to the exceptions listed here, aluminum and iron spikes are not required for soil samples.
- b) If the control criteria given in section (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. For unfiltered water samples and solid samples that have not been crushed and homogenized, results associated with a failed MS analysis may be qualified and reported without reanalysis.
- c) For ion chromatography, 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 in section (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 (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 four 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 CRI and CRA analyses

CRA (AA) and CRI (ICP-AES and ICP-MS) standards are run at the beginning of each analysis run as a measure of accuracy near the reporting limit. CRA standards are prepared with concentrations at approximately the PQL, and CRI standards are at approximately twice the PQL. If the low calibration standard is run at a level appropriate to the CRI, 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 CLP Form II (Part 2) or equivalent. The acceptance criterion for these analyses is 70 to 130 percent recovery. If the CRI or CRA recoveries are outside the control limit, the chemist must terminate the analysis, initiate corrective action, and successfully reanalyze the CRI or CRA before proceeding.

#### 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, including the control criteria for internal standard intensities given in section 8.3 of method 6020, shall be followed.

### 3.5 Organic analytical and QC requirements

Organic analytical and quality control requirements are specified in this section of the SOW. The laboratory shall follow the requirements specified in USEPA SW-846 or other EPA methods as requested by the DOE-AL facility SMO. Additional general analytical and quality control criteria are specified in the sections 3.5.1 through 3.5.10. Method-specific analytical requirements are given in section 3.5.11. The DOE-AL facility'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, GC, GC/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 SW-846 method. All initial calibrations will be verified using a second-source calibration verification standard. Laboratories may a) verify the calibration using a second-source standard immediately after the initial calibration, b) use a second-source CCV standard with each analytical run, or c) use a second source LCS. If a second source LCS is used, the LCS must contain all of the compounds in the initial calibration. *Note for this issue that most DOE-AL facilities now require full list LCS samples to be run and reported for all or most of their work. Laboratories should obtain contract-specific guidance on this issue from the individual facility SMOs.*

a) Calibration acceptance criteria

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 initial calibration standards should be less than 20 percent unless otherwise specified in the method. RSD averaging, as discussed in SW-846 method 8000, may be used for GC/MS methods but shall not be used for GC or HPLC methods. If RSD averaging is used, no individual RSD should exceed 30 percent without prior approval from the DOE-AL facility. If the initial calibration fails these criteria, the laboratory shall take corrective action and perform a new initial calibration.
- ii. The percent difference of the daily or continuing calibration standard RF from the average RF obtained from the initial calibration must be within  $\pm 15$  percent unless otherwise specified in the method. CCV recovery averaging, as discussed in SW-846 method 8000, may be used for GC/MS methods but shall not be used for GC or HPLC methods. If recovery averaging is used, no individual recovery should exceed a  $\pm 60$  percent control criterion. If calibration verifications fail these criteria, the laboratory shall take corrective action and perform a new initial calibration. As specified in SW-846 method 8000, 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.

Note: CCV averaging need only include the compounds that are to be reported. Laboratories need not include all the compounds from the initial calibration if some are not requested as target analytes. However, compounds that are not considered in the

CCV average must be listed in the case narrative as having been excluded.

- iii. As described in SW-846 method 8000B, laboratories may use least-squares regression to generate linear calibration curves, provided the correlation coefficients for the resulting curves are at least 0.99. 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 allowable magnitude of the Y-intercept.

b) Low-concentration soil VOC analysis calibration

For SW-846 methods 8021B and 8260B, 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 DOE-AL facility SMO 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 DOE-AL facility SMO 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) VOC analysis

For SW-846 methods 8021B and 8260B, water and soil samples shall be prepared and purged into the GC instrument using method 5030B or 5035, as appropriate to the sample vessel provided and individual DOE-AL facility contract requirements. **(Insert your requirements here.)** The laboratory may employ the VOC screening procedures described in SW-846 methods 3810 or 3820, to determine whether sample dilution is required.

b) VOC 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 8260B shall require a medium-concentration soil analysis. The smallest amount of soil sample on which a low-concentration analysis shall be performed is one gram. Medium and high-concentration soil and solid waste samples shall

be extracted using methanol as described in method 5035.

c) TCLP VOC extractions

For VOC TCLP 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," to extract samples.

d) Sample extraction and cleanup

Depending upon the characteristics of the sample matrix, the laboratory shall use an appropriate SW-846 3500 series method to extract samples for SW-846 methods 8081A, 8082, 8141A, 8151A, and 8270C.

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.

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 DOE-AL facility.

- a) Sample extraction and analysis, confirmation of detection, and any required re-analyses, 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 8000B. The retention times for all analytes in all CCVs must be within the retention time windows established with the initial calibration.
- c) Surrogate recovery acceptance criteria should be calculated according to the guidance given in SW-846 method 8000B. 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 non-routine 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.

- 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. Dilutions performed because one or more analytes exceeded the high calibration standard shall be done in such a way as to ensure that the resulting concentration for each target analyte that exceeded the calibration range is greater than the PQL.
- f) The additional method-specific sample analysis acceptance criteria given in section 3.5.11 must be met.

#### 3.5.6 Blank analysis

##### a) Method blank (preparation blank) analysis

The laboratory shall run a method blank 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. Method blanks 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 8081A, 8141A, 8151A, and 8270C, soil method blanks shall consist of 30 grams of reagent anhydrous sodium sulfate that has been taken through the same preparation steps as those used for the samples. Method blank acceptance criteria are given below in section (d). Samples associated with an unacceptable method blank analysis shall be reanalyzed at no additional cost to the DOE-AL facility.

##### b) Instrument blank analysis

A blank shall be run after 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 section (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 always possible to insert blanks for automated analytical runs. If blanks were not run, the sample immediately following the contaminated sample shall be re-analyzed at no additional cost to the DOE-AL facility if

it shows the same compound(s) that were over the calibration range in the high sample.

c) Storage blank analysis

For SW-846 methods 8021B and 8260B, laboratories shall prepare one storage blank sample for each week in the month, at the beginning of each month, 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 DOE-AL facility SMO 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 DOE-AL facility 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 PQL. This blank acceptance criterion is waived in two cases; when DOE-AL facility samples show no target analyte "hits" on compounds detected in the blank, and when the associated sample results are  $\geq 10X$  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 ten times the concentration in the blank. All samples having that compound's concentration at less than ten 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 DOE-AL facilities 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. If solvent blanks are run immediately before QC samples in isolated cases, the reasons for this must be discussed in the associated instrument run logs.

3.5.7 MS and matrix spike duplicate (MSD) analyses

- a) The laboratory shall perform MS and MSD analyses for all methods except 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 DOE-AL facility 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 8000B and the QC acceptance criteria found within the specific SW-846 method. Laboratories will report recoveries and RPD values for MS and MSD analyses in the QC section of deliverables.
- c) Laboratories shall use the spiking compounds specified in SW-846 as appropriate to the method. Where no spiking compounds are specified in the method, laboratories shall use a representative subset of the target compounds provided that at least one MS compound is quantified against each internal standard.

3.5.8 LCS 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 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 8000B 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 DOE-AL facility.

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). If the LCS is not used for second-source calibration verification, "short-list" LCS compounds from the same source as the calibration standards may be used provided that at least one LCS compound is quantified against each internal standard. For short list LCS samples, all compounds must pass the established acceptance criteria. For full-list LCS samples, see (e) below. 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 LCS acceptance windows must not exceed MS acceptance windows.
- c) For non-routine analytes, the recovery control limits shall not be less than 10% and not be greater than 150%.
- d) If the LCS fails, corrective action (re-extraction and reanalysis) must be performed regardless of the outcome of the MS and MSD analyses.
- e) If a full-list LCS is used, up to 5% of the analytes may fail the LCS acceptance criteria without initiating corrective action. All LCS analyte failures MUST be documented and discussed in the applicable case narrative(s).

#### 3.5.9 Second-column or GC/MS 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 (8330 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%, 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 DOE-AL facility samples will 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 compounds listed in the attachments for the methods below are those listed as possible in the methods. The actual target analyte list to be used should be negotiated by each DOE-AL facility based upon site needs.**

##### a) Petroleum hydrocarbons by GC/Flame Ionization Detector (FID)

Unless otherwise specified by the DOE-AL facility, petroleum hydrocarbon analysis shall be performed using a modified SW-846 method 8015B. Regardless of the method specified, laboratories shall adhere to the QC requirements given in this SOW, SW-846 method 8000B, and SW-846 method 8015B. Soil samples shall be extracted using methanol, methylene chloride, or other appropriate solvents. Water samples for volatile petroleum hydrocarbons will be purged. Analysis will be via a GC/FID instrument. At the request of the DOE-AL facility SMO, the instrument may be calibrated for petroleum hydrocarbons based on a range of molecular weights or product type (such as gasoline range organics), 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. Instrument calibration shall be based on the integrated area for all petroleum hydrocarbon peaks present in standard chromatograms.
- ii. Analysis of a blank is required at least once in each 12-hour period.
- iii. Modified 8015B method analyses are exempt from the sample acceptance criteria requiring extract cleanup and reanalysis based upon surrogate recovery.
- iv. When an unusually high concentration sample is encountered, it should be followed by a solvent blank to check for carryover contamination.

- v. The concentrations of the LCS compounds shall be near the midpoint of the calibration range. The laboratory shall calculate data acceptance criteria using the procedure for QC check samples given in SW-846 method 8000B.

b) Organochlorine pesticides by GC

Unless otherwise specified by the DOE-AL facility, organochlorine pesticide analysis shall be performed according to the requirements listed in SW-846 methods 8081A, "Organochlorine Pesticides by Gas Chromatography." Regardless of the method specified, laboratories shall adhere to the QC requirements given in this SOW, SW-846 method 8000B, and SW-846 method 8081A. **(It is recommended that the DOE-AL facility use method 8081A in place of method 608. The target analyte list for 608 is a subset of the TAL for 8081A. However, the analytical and QC criteria are more clearly defined in 8081A.)**

- i. All soil sample extracts shall be subjected to the Florisil cartridge cleanup procedure described in method 3620B. 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. A blank consisting of clean solvent containing only the surrogate compounds shall be analyzed at the beginning and end of each analytical run, and once every 20 analytical samples. If the system is primed prior to analysis, the initial blank should be run before any standards or samples. Subsequent blanks in the analytical run should follow the CCVs.
- v. The following additional sample analysis acceptance criteria are required:
  - 1. The laboratory shall derive surrogate recovery control limits according to the procedure given in SW-846 method 8000B
  - 2. A CCV for any identified multi-component analyte 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.

3. 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.
  4. Confirmation of all target analyte “hits” 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%, the lower of the two shall be reported on the Form I.
- vi. A CCV must be run at the beginning of each analytical run, at least once every 20 samples (preferably every 10), and at the end of each analytical run (see SW-846 method 8000B, section 8.2.2 for bracketing requirements). Recovery for the CCV target compounds must be within  $\pm 15$  percent. If the CCV fails this acceptance criterion, analysts must take corrective action and reanalyze all extracts run since the last successful CCV analysis.
  - vii. If an internal standard is used, the internal standard area criteria and corrective actions specified in section 8.4.5 of method 8081A apply.
  - viii. 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 for the DOE-AL facility, an LCS shall be prepared and run for an 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 8000B.
- c) Polychlorinated biphenyls (PCBs) by GC

Unless otherwise specified by the DOE-AL facility, PCB analysis shall be performed according to the requirements listed in SW-846 method 8082, “Polychlorinated Biphenyls by GC.” Regardless of the method specified, laboratories shall adhere to the QC requirements given in this SOW, SW-846 method 8000B, and SW-846 method 8082.

- i.  $\text{H}_2\text{SO}_4/\text{KMnO}_4$  cleanup (method 3665A) is strongly recommended for all sample extracts. Modification of the cleanup procedure to neglect the  $\text{KMnO}_4$  step is acceptable, provided that  $\text{H}_2\text{SO}_4$  alone produces acceptable results. However, laboratory SOPs should

provide instruction on the  $\text{KMnO}_4$  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. DOE-AL facilities may request that laboratories 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 PCBs are determined as Aroclors, decachlorobiphenyl shall be added to each sample extract as a surrogate.
- v. A CCV must be run at the beginning of each analytical run, at least once every 20 samples (preferably every 10), and at the end of each analytical run. Recovery for the CCV compounds must be within  $\pm 15$  percent. If the CCV fails this acceptance criterion, analysts must take corrective action and reanalyze all extracts run since the last successful CCV analysis.
- vi. The internal standard area criteria and corrective actions specified in section 8.3.3 of method 8082 apply.
- vii. LCS analyses are required at a frequency of one per batch or one per 20 samples, whichever is more frequent. LCS data acceptance criteria shall be derived by the laboratory according to the procedure for QC check samples given in SW-846 method 8000B.
- viii. 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.
- ix. Confirmation of all target analyte "hits" 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%, the higher of the two shall be reported on the Form I.
- x. If an initial calibration has not been performed for any target Aroclor for which a "hit" above the MDL is obtained, laboratories 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 "hit" above the MDL is obtained, an

acceptable CCV run must be obtained within 72 hours, at minimum, if the result is to be reported.

d) Chlorinated herbicides by GC

Unless otherwise specified by the DOE-AL facility, chlorinated herbicide analysis shall be performed according to the requirements listed in the SW-846 methods 8151A, "Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzoylation Derivatization: Capillary Column Technique." Regardless of the method specified, laboratories shall adhere to the QC requirements given in this SOW, SW-846 method 8000B, 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 samples as the surrogate compound. In this case, the laboratory should consult the DOE-AL facility on the selection of the surrogate compound.
- ii. A CCV must be run at the beginning of each analytical run, at least once every ten samples, and at the end of each analytical run. Recovery for the CCV compounds must be within  $\pm 15$  percent. If the CCV fails this acceptance criterion, analysts must take corrective action and reanalyze all extracts run since the last successful CCV analysis.
- iii. The LCS shall contain each of the specified target chlorinated herbicides. The laboratory shall derive data acceptance criteria using the procedure for QC check samples given in SW-846 method 8000B.
- iv. 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.
- v. Confirmation of all target analyte "hits" 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%, the higher of the two shall be reported on the Form I.

e) VOC analysis by GC/MS

Unless otherwise specified by the DOE-AL facility, VOC analysis shall be performed according to the requirements listed in the SW-846 method

8260B, "VOCs by GC/MS". Regardless of the method specified, laboratories shall consider the QC requirements given in the SOW, SW-846 method 8000B, and SW-846 method 8260B 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 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. As stated in method 8260B, the minimum average RF for the initial calibration's system performance check compounds (SPCCs) shall be 0.10 for chloromethane, bromoform, and 1,1-dichloroethane, and 0.30 for 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum average RF for all other calibration compounds should be 0.01. These criteria also apply to daily CCV standards.
- ii. A CCV, followed by a method blank, shall be run at least once every 12 hours. A method blank will also be run immediately after the initial calibration standards. A known value  $\pm 15$  percent recovery control criterion applies to all target compounds in the CCV. However, for GC/MS methods, CCV recovery averaging is allowed. If CCV averaging is used, the average recovery for all target compounds must be within  $\pm 15$  percent, no individual recovery value shall exceed  $\pm 60$  percent control criteria, and each individual target compound that recovers outside the  $\pm 15$  percent window must be called out in the case narrative.
- iii. 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 five times the their required PQL. If these concentration limits are exceeded, laboratories shall discuss the blank contamination in the associated case narrative.
- iv. No quantitation ion may saturate the instrument's detector. Blank analyses must follow immediately when this occurs, and additional decontamination procedures must be employed as necessary. If a blank has not been run because the analytical run was automated, the subsequent sample must be reanalyzed if it shows the same compound(s) that were over the calibration range in the previous sample.
- v. An LCS must be included in each analytical batch. See sections 3.5.2 and 3.5.8 of this SOW for LCS requirements. The laboratory shall calculate percent recovery acceptance criteria by the using the procedure for QC check samples in SW-846 method 8000B.
- vi. 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 ten 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.

- vii. The linearity requirements of method 8260B, section 7.3.8, apply (RSD for target analytes and calibration check compounds [CCCs] should be 15% or less). Refer to SW-846 method 8000B for RF RSD averaging, and section 3.5.2 of this SOW for least-squares regression calibration options. If RF RSD averaging is done, every compound that fails the 15% RSD control criterion must be called out in the applicable case narrative. In addition, no CCC can exceed a 30% RSD acceptance criterion, and no other target compound can exceed the 30% criterion without prior approval from the DOE-AL facility SMO.
- viii. The acceptance criteria (80 to 120 percent recovery) and corrective actions (recalibrate if the problem cannot be corrected) for CCCs given in section 7.4.5 of the method shall be strictly followed. The CCCs are listed in section 7.3.6.3 of the method. These control criteria apply to the CCCs in both the initial and the CCV analyses. In addition, the control criteria for the CCCs supersede the CCV
- ix. 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).
- x. Per section 7.4.7 of 8260B, if the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

f) SVOC analysis by GC/MS

Unless otherwise requested by the DOE-AL facility, SVOC analysis shall be performed according to the requirements listed in the SW-846 method 8270C, "SVOC by GC/MS." Regardless of the method specified, laboratories shall consider the QC requirements given in the SOW, SW-846 method 8000B, and SW-846 method 8270C to be the minimum requirements.

- i. For soil, sediment, and biological samples, GPC cleanup using SW-846 method 3640A 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 method 8270C, the minimum average RF for the initial calibration's SPCCs shall be 0.05. The SPCCs for 8270C are N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. The minimum average RF for all other calibration compounds should be 0.01. These criteria also apply to daily CCV standards.
- iii. A CCV, followed by a method blank, shall be run at least once every 12 hours. A method blank will also be run immediately after the initial calibration standards. A known value  $\pm 15$  percent recovery control criterion applies to all target compounds in the CCV. However, for GC/MS methods, CCV recovery averaging is allowed. If CCV averaging is used, the average recovery for all target compounds must be within  $\pm 15$  percent, no individual recovery value shall exceed  $\pm 60$  percent control criteria, and each individual target compound that recovers outside the  $\pm 15$  percent window must be called out in the case narrative.
- iv. The target phthalate esters are exempt from the reanalysis requirements associated with method blank contamination up to a concentration of five times the PQL.
- v. No quantitation ion may saturate the instrument's detector. Solvent blank analyses must follow immediately when this occurs, and additional decontamination procedures must be employed as necessary. If a solvent blank has not been run because the analytical run was automated, the subsequent sample must be reanalyzed if it shows the same compound(s) that were over the calibration range in the previous sample.
- vi. An LCS must be included in each extraction batch of up to 20 samples. See sections 3.5.2 and 3.5.8 of this SOW for LCS requirements. The laboratory shall calculate percent recovery acceptance criteria by the using the procedure for QC check samples in SW-846 method 8000B.
- vii. 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 ten 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.
- viii. The linearity requirements of method 8270C, section 7.3.7, apply (RSD for target analytes and CCCs should be 15% or less). Refer

to SW-846 method 8000B for RF RSD averaging, and section 3.5.2 of this SOW for least-squares regression calibration options. If RF RSD averaging is done, every compound that fails the 15% RSD control criterion must be called out in the applicable case narrative. In addition, no CCC can exceed a 30% RSD acceptance criterion, and no other target compound can exceed the 30% criterion without prior approval from the DOE-AL facility SMO.

- ix. The acceptance criteria (80 to 120 percent recovery) and corrective actions (recalibrate if the problem cannot be corrected) for CCCs given in section 7.4.5 of the method shall be strictly followed. The CCCs are listed in Table 4 of the method. These control criteria apply to the CCCs in both the initial and the CCV analyses. In addition, the control criteria for the CCCs supersede the CCV averaging acceptance criteria given in item (iii) above.
- x. 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 TICs.
- xi. Per section 7.4.7 of 8270C, if the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

g) Polychlorinated dioxins and furans by GC/MS

Unless otherwise specified by the DOE-AL facility, sample analysis shall be performed according to the requirements listed in the SW-846 method 8280A, "The Analysis of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans by High Resolution GC/Low Resolution MS.

Method 8280A is intended for low resolution work. The high resolution method (8290) is considered to be a "special analytical service" and is procured separately from this DOE-AL facility contract.

- i. The suggested QC analyses in method 8280A, including LCS, MS/MSD, and blank analyses, are required under this SOW.
- ii. Laboratories shall follow the method 8280A calibration, analysis, and analyte identification specifications given in sections 7 and 8 of the method.

h) Nitroaromatics and nitramines by HPLC

Nitroaromatics and nitramines analysis shall be performed according to the requirements specified in the SW-846 method 8330, "Nitroaromatics and Nitramines by HPLC." Laboratories shall adhere to the QC requirements of this SOW, SW-846 method 8000B, and SW-846 method 8330.

- 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, we recommend that either 1,2-dinitrobenzene or 1,4-dinitrobenzene be used as the surrogate.
- ii. A CCV must be run at the beginning of each analytical run, at least once every 10 samples, and at the end of each analytical run. Recovery for the CCV target compounds must be within  $\pm 15$  percent. If the CCV fails this acceptance criterion, analysts must take corrective action and reanalyze all extracts run since the last successful CCV analysis.
- iii. The method blank should be run immediately after the calibration verification (CCV) sample. A blank shall be run at least once in each 12-hour period.
- iv. The LCS shall contain all of the target analytes at concentrations near the midpoint of the calibration range. The laboratory shall statistically derive the acceptance criteria for LCS compounds according to the guidance provided in SW-846 method 8000B.
- v. Appropriately scaled chromatograms shall be provided for all dilutions for which data are reported. Laboratories shall use run times that are adequate to achieve reasonable peak resolution; compressed runs that cause unnecessary co-elutions must be avoided.
- vi. All target analyte "hits" in field samples shall be confirmed on a CN column. The estimated analyte value, obtained using at least one standard to calibrate the CN column, shall be reported for confirmation analyses. Coelutions are prohibited in CN column calibrations. Laboratories shall only report the results obtained from the C18 column on the Form I, regardless of RPD between the C18 and CN column results.
- vii. Solid phase extraction (SPE) has not yet been generally approved for HE extractions in low-level waters in DOE-AL facility work. Laboratories shall use the method specified salting out procedure until matrix-specific method efficacy studies for SPE are submitted to and approved by the DOE-AL facility.

- viii. Laboratories shall analyze and report CCVs and method blanks in CN column confirmation runs. The analysis frequency requirements in sections (ii) and (iii) above shall be met.
- ix. Limited spectral information is available if a diode array detector is used. This approach is preferred by the DOE-AL facility SMOs because of the increased ability to identify false positives that it affords. However, this does not replace the requirement for CN column confirmation of target hits.
- x. The comments of this section apply to C18 hits that have been confirmed on the CN column. If LC/MS/MS is to be used as an additional confirmation for HPLC 8330 target analyte detections, the instrument will be calibrated according to the guidance in methods 8321A and 8000B. An exception to this is calibration frequency, where 8000B will supercede. Recalibration is required when ion sources are changed and when CCV results do not pass the  $\pm 15$  percent (average) acceptance criterion. Calibration will include a minimum of a blank and five standards. The initial calibration curve is subject to the 20 percent RSD criterion given method 8000B.
  - 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  $\pm 30$  percent of the average internal standard areas from the initial calibration.
  - 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. Calibration verification and instrument blank samples (CCV and CCB) shall be analyzed at the beginning of each analytical run, at least once every ten 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  $\pm 15$  percent. Averaging the recoveries of the target compounds is acceptable, provided that no recovery for any single compound exceeds 20 percent difference from the known value.
  - 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 preparation blank 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. **(Insert your facility's analytical spike requirements here.)**
  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 analyte 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 CN columns to be reportable detections in 8330 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 8330 that are given in Attachment 5 of this document 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 methods that do not monitor such transitions shall not be used.
- xi. If HE compounds are to be run by LC/MS/MS as the primary detection technique, then the internal standard, calibration, MDL, and calibration verification requirements of section (x) above apply. In this work, laboratories shall use same extraction procedures that are used for 8330 HPLC analyses.
1. Surrogate recovery, matrix spike, and LCS acceptance criteria shall be separately established via statistical process control for HE by LC/MS/MS. The LCS and matrix spikes shall contain the full list of target analytes.
  2. Matrix spikes shall be prepared at concentrations between five and ten times the concentration of the MDL values for each target analyte, and shall be analyzed at

least once per batch.

3. The analytical spikes discussed in (x)(5) should be run at least until the reliability of the method is thoroughly demonstrated.
4. All holding times for 8330 that are given in Attachment 5 of this document apply.
5. As in the confirmation protocols discussed in (x) above, only LC/MS/MS methods are acceptable for this work

i) VOCs in ambient air using GC

Analysis shall be performed according to the requirements specified in EPA method TO-14A, "Determination of VOCs in Ambient Air Using SUMMA<sup>®</sup> Passivated Canister Sampling and Gas Chromatographic Analysis," 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 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. For GC/MS analytical systems, the GC/MS system tune check shall be performed daily prior to sample analysis by sampling a canister containing BFB. The BFB tune check mass spectrum shall meet the ion abundance criteria listed in Table 4 of the method. If the relative abundance for any of the ions listed Table 4 differs by  $\geq$  ten percent from those obtained during the previous day's BFB tune check, the instrument must be re-calibrated.
- v. Initial instrument calibration shall be performed using three standard concentration levels and a humid zero air standard. Daily calibration verification shall be performed using a mid-range standard. Recoveries for the target analytes in the calibration verification standard must be within  $\pm$  20 percent of the known values.
- vi. As discussed in the method, the retention time windows for all target analytes must be checked via the analysis of three standards at least once every 72 hours.
- vii. As discussed in the method, the retention time for compounds identified in samples must be within  $\pm$  0.1 minutes of the

theoretical retention times resulting from the calibration or retention time check runs. In addition, the relative abundance of quantitation ions must be within  $\pm 15$  percent of those in the applicable reference mass spectra. Exceptions to this are vinyl chloride and methylene chloride, for which the acceptance criterion is  $\pm 25$  percent.

- viii. MS and MSD are not required for this method.
- ix. The LCS shall contain all of the target analytes at concentrations near the midpoint of the calibration range. Recovery for the target analytes must be within  $\pm 20$  percent of the theoretical value.
- x. Laboratories shall use a minimum of three surrogate compounds and report the resulting surrogate recovery data with TO-14 QC deliverables.

### 3.6 Radiochemistry analytical QC requirements

Standards used in batch quality control analyses, such as LCS and spiking standards, need not be NIST traceable. Standards requiring NIST traceability are discussed in the calibration 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. PB results shall be calculated assuming aliquot sizes comparable to the sample aliquots used in the analysis.
- b) If any blank result is greater than its associated DLC, the case narrative for that deliverable shall specifically discuss that fact.
- c) Samples associated with any PB result that is greater than its associated MDC shall be redigested and reanalyzed. Exceptions to this requirement are samples for which the measured concentration is greater than or equal to five times the PB value. Reanalysis is not required for such samples.
- d) 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 DOE-AL facility 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 DOE-AL facility SMO or the DOE-AL Analytical Management Program (AMP) staff 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 DOE-AL facility SMO or AMP can obtain appropriate solid LCS materials.
- b) The aqueous LCS analytical results shall agree within  $\pm 20$  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 quality control data for gamma spectroscopy shall include Am-241 (59.5 keV), Cs-137 (661.7 keV), and Co-60 (1332 keV) at minimum.

### 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:

$$\text{RER} = \frac{|S - R|}{\sigma_{95S} + \sigma_{95R}}$$

- where, RER = replicate error ratio  
S = sample value (original)  
R = replicate sample value  
 $\sigma_{95S}$  = sample uncertainty (95%)  
 $\sigma_{95R}$  = replicate uncertainty (95%)

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 one. 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 MDCs.

- b) If the RER control criterion is 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 (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 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 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 DOE-AL facility SMO 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 section (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) MSs 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 <sup>3</sup>H and <sup>99</sup>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 not exceed 100 pCi/L or 100 pCi/g. Tritium samples must be spiked before the distillation step, at a level chosen by the laboratory.
- d) The spike recovery control limits are  $\pm 25$  percent. An exception to these control limits is made in the case for which the sample result exceeds four 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 DOE-AL facility SMO has given prior approval for a batch-correction procedure.
- b) Recovery guidelines for tracer and carrier results in routine matrices (soil and water) shall be 50 to 105 percent. The DOE-AL facility SMO 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 DOE-AL facility SMO 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 DOE-facility SMO 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 met, then the DOE-AL facility SMO should approve reporting the results without reanalysis.

- c) The concentration of tracer material added shall be sufficient to result in a maximum of five 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 MDCs

The tables in Attachment 2 give target MDC values for radionuclide analyses by analytical technique and matrix. Laboratories shall adjust analytical conditions to meet the target MDCs. **(Insert your target MDCs in the tables.)**

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 MDCs for those radionuclides. The analytical conditions chosen will determine the MDCs for other reported nuclides.

#### 3.6.9 Counting instrument calibration requirements

Radionuclide analyses that do not involve nuclear disintegrations are defined to be general inorganic analyses. Such analyses are subject to the analytical and quality control requirements in the appropriate section of this SOW. This applies to uranium determination by fluorimetry and ICP-MS, as well as any radionuclide determinations by column-extraction/flow-injection ICP-MS. Radiochemistry counting instruments are subject to the minimum calibration requirements given below.

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 DOE-AL facility SMO if corrections are made.

a) Gas flow proportional counting

- i. Background counts equal in duration to the longest expected sample count time shall be performed at least monthly.
- ii. Daily background checks shall be performed.
- iii. Detector efficiency shall be determined at least annually, with recalibration 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 least annually, based upon the acquisition of at least 10,000 counts for each calibration standard.
- vi. Voltage plateau curve determinations shall be made at least annually, with performance checks made after each gas bottle change or maintenance activity.
- vii. Crosstalk determinations shall be made at least annually.
- viii. Laboratory calibration procedures shall require that backgrounds be checked after counting high-activity samples.

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 performed at least annually, with verification performed at least monthly.
- iii. Detector efficiency shall be determined at least annually, with verification performed 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 method blank for weekly background checks is acceptable, provided that the data are compared to the original calibration background.
  - ii. Energy/channel calibrations shall be performed at least annually, with verification performed weekly.
  - iii. Efficiency calibrations shall be performed at least annually, with verification performed weekly.
  - iv. Resolution calibrations shall be performed annually, with verification performed weekly.
- d) Liquid scintillation
- i. Efficiency/quench curves shall be established at least annually for each radionuclide to be counted, with daily verification checks performed.
  - ii. The vendor-supplied set of calibration vials shall be run with each sample batch.
  - iii. Each batch shall contain a calibration blank vial to be used for blank subtraction (see section 3.6.7 of this SOW).
- e) Kinetic phosphorescence analysis (KPA) for uranium
- KPA has been found to be unreliable due to strong susceptibility to interferences from constituents commonly found in DOE-AL facility samples. KPA shall not be used in the analysis of DOE-AL facility samples. ICP-MS is the preferred technique for total uranium determinations; however, fluorimetry may also be used.
- 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 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 MDC that are identified using the gamma spectroscopy software without any additional charge to the DOE-AL facility.

#### 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 DOE-AL facility 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.

### 3.7 Asbestos analysis

#### 3.7.1 Accreditation

Laboratories must be accredited by the AIHA to be eligible to perform airborne asbestos analysis for the DOE-AL facility. Laboratories must participate in and report results to the DOE-AL facility SMO for all PE rounds to demonstrate that the accreditation is current. In addition, the DOE-AL 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 DOE-AL facility SMO.

### 3.8 Geotechnical analyses

#### 3.8.1 Accreditation

Laboratories performing geotechnical analyses shall have current accreditation under the American Association of State Highway Transportation Officials (AASHTO) accreditation program.

#### 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 quality control 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

Some laboratories will wish to use CLP software to generate forms for certain analyses. Information required in the sections below that cannot be accommodated by the CLP software may be provided on attached forms where necessary. **DOE-AL facilities should discuss the required site-specific reporting units for general inorganic chemistry, organic chemistry, and radiochemistry here.**

#### 4.1.1 Level C analytical reports

Level C analytical data packages provided to the DOE-AL facility SMO shall

contain all the items discussed in sections (a) through (f), below. One photocopy of the hard copy deliverable is required with each submittal. A comprehensive case narrative is required for all data reports submitted.

- a) A completed Deliverable Transmittal/Review form.
- 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 DOE-AL facility 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 (Form I) for each sample associated with the deliverable. The required contents of each Analysis Results form are outlined in section 4.1.3 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.
- e) Signed and dated original COC forms received with each sample shipment, indicating sample receipt and custody by the laboratory.
- f) One EDD copy of the analytical data and quality control results formatted as outlined below in section 4.1.6 of this SOW.

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.

#### 4.1.2 Level D analytical reports

- a) Level D analytical reports shall include all materials required in Level C above, shipping and login documents, 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 shipping documents, 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 recreate 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 analyst worksheets shall include certification that the required calibrations were successfully performed on schedule and reference the relevant computer file names and dates.

- 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.
- d) Level D deliverables shall be on 8½- by 11-inch paper, one-sided, and paginated

#### 4.1.3 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 preferred, although these must be adapted for use with non-CLP methods. This decision is left to the discretion of the laboratory.
- b) The laboratory shall specify the DOE-AL facility sample ID, date analyzed, date extracted (where appropriate), delivery order number (SDG field), report date, 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 MDC and DLC (as applicable), units of measure, data qualifier(s), method of analysis, 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.

#### 4.1.4 "Less than" results

Mathematical "less than" signs shall not be used in reporting DOE-AL facility 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 MDC or DLC shall be reported as measured, as discussed in section 3.3.4 of this SOW.

#### 4.1.5 Analytical uncertainties and detection limits

The analytical uncertainty values and MDCs for radiochemical parameters shall be reported with each result on both the hard copy and the EDD.

#### 4.1.6 EDD format

Format requirements for the EDD copy of analytical data are provided as **Attachment XX**. The data for the hard copy deliverable and the EDD shall be drawn from the same database at the same time.

- a) The laboratory shall have 60 calendar days to comply with any changes made to the EDD specification after the contract is awarded.
- b) Expenses incurred by the laboratory in implementing changes made to the EDD specification after the contract is awarded will be reimbursed at cost.
- c) The laboratory shall have **XX** calendar days from the contract award date to develop compliant EDDs. **Insert your contract requirement for time to develop EDDs here.**

#### 4.1.7 Reporting conventions

Anion reporting conventions are as listed below:

- a) Ammonium is reported as N.
- b)  $\text{NO}_2 + \text{NO}_3$  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  $\text{SO}_4$ .
- g) **Modify the conventions above or insert additional ones as necessary for your facility.**

#### 4.1.8 QC 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, and the use of "CLP like" tabular forms is preferred.
  - 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. Provide analysis run logs, EPA CLP Form XIV-IN or equivalent for all parameters.
- b) QC data deliverables for radiochemistry 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, and the use of "CLP like" tabular forms is preferred.
- 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.
  - 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, record of SMO 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, and the use of tabular "CLP like" forms is preferred.
- i. Initial calibration data, ICV data, and CCV data shall be presented. The initial calibration data shall include the average RF and RSD, or the curve equations and correlation coefficients if regression is used. The calibration verification data shall include the percent recovery 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 DOE-AL facility on a 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 a qualifier is required by the DOE-AL facility.
- 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 qualifier is required by the DOE-AL facility.
- 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 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.

#### 4.1.9 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, N," and "\*" may be used in reporting radionuclide and gross radiation results. **(Some qualifiers, such as the "H" and "I" flags, must be changed to suit specific facility needs. Additional qualifiers may also be needed for radiochemistry, such as a "BD:" flag for results below the MDC or a flag for low tracer recovery.)**

- a) In the event that the holding time for a particular parameter had expired prior to analysis, flag the associated results with an "H."
- 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) GFAA analytical results associated with a post-digestion spike that was outside the control limits, while the sample absorbance was less than 50 percent the spike absorbance, shall be qualified with the "W" flag. The use of this data qualifier is discussed in the CLP SOW.
- g) GFAA analytical results associated with a duplicate injection that failed to meet the duplicate injection precision criterion shall be qualified with the "M" flag. The precision criterion for use of this data qualifier is discussed further in the CLP SOW.
- h) GFAA analytical results obtained by the method of standard additions (MSA) shall be qualified with the "S" flag.
- i) GFAA analytical results obtained by MSA, and for which the MSA correlation coefficient is less than 0.995, shall be qualified with the "+" flag.
- j) General inorganic results having concentrations between the MDL and the PQL (or reporting limit) shall be qualified with a "J" flag.
- k) 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.

#### 4.1.10 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.
- 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) The “N” flag indicates presumptive evidence of a compound. This flag is typically only used for TICs, where the identification is based on a mass spectral library search. However, it may also be used when a compound is detected on a primary column and cannot be confirmed because of the presence of an interference on the confirmation column. This latter case must be discussed in the associated case narrative. The “N” flag is applied to all TIC results. For generic characterization of a TIC, such as chlorinated hydrocarbon, the N flag is not used.
  - 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 Form I for the original analysis.
  - f) If a sample or extract is reanalyzed at a higher dilution factor, for example when the concentration of an analyte exceeds the upper calibration range, the DL suffix is appended to the sample number for the more diluted sample.
  - g) In the event that the required holding time to extraction or holding time to analysis was missed, flag the associated results with an "H."
  - h) The “X” qualifier is used only to denote the existence of presumptive evidence suggesting that the reported analyte is not present in the sample. That is, this qualifier may be used only to indicate that the chemist believes the result to be a false positive. When the “X” qualifier is used, laboratories must provide supporting data and explanatory case narrative comments in the data package.

#### 4.1.11 Completeness

Partial deliverables shall not be submitted to the DOE-AL facility SMO unless specifically requested. In addition to the deliverable requirements given in this section, the DOE-AL facility SMO 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 DOE-AL facility 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 DOE-AL facility SMO.

#### 4.1.12 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, or MDCs in the attachments.

#### 4.2 Analytical data deliverable deadlines

##### 4.2.1 Turn-around times

- a) A report of analytical results is due to the DOE-AL facility SMO 30 calendar days from the date of receipt of the last sample associated with each delivery order when standard turn-around time is requested. Turn-around times for accelerated delivery requests shall be 1, 3, 5, 10, and 15 working days, and shall be mutually agreed upon by laboratory staff members and the DOE-AL facility SMO. Reports with accelerated turn-around times shall be faxed to the DOE-AL facility SMO, in the laboratory's LIMS format if desired, with the full deliverable due at the normal 30-day deadline.

- b) Corrected reports and reports for any requested reanalyses are due ten 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 DOE-AL facility SMO reserves the right to request expedited reanalyses when circumstances require this. Reimbursement shall be made according to the specifications of section 1.7.2 of this SOW, and will be at the standard turn-around time rates unless expedited reanalyses are requested. For reanalysis turn-around times less than ten working days, payment will be at the applicable rate for the corresponding expedited analyses (subject to the stipulations of section 1.7.2). Reanalysis reports shall be submitted according to the guidelines for the report level originally requested for that delivery order.
- c) Invoices shall be submitted monthly for the delivery orders reported in that period. Invoices shall contain delivery order numbers, the number of samples, analyses performed, unit cost, and extended cost. Invoices shall be itemized and organized in such a way as to facilitate detailed review and cost verification without additional laboratory input.

#### 4.2.2 Level D report deliverable deadlines

Level D deliverables are typically required. However, if Level C reports are the requested deliverable, 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 DOE-AL facility SMO.
- b) When Level D deliverables are requested to support data that have been delivered previously, the Level D deliverable shall be due two 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 DOE-AL facility SMO. Price reductions may be imposed for late deliverables at the discretion of the DOE-AL facility SMO, depending on the contributing circumstances, at the rate of two percent per working day.
- 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 turn around time. That is, a report for results with a ten-day requested turn around that arrives on the 15<sup>th</sup> day will be paid for at the 15-day turn around rates less ten percent.
- c) Price reductions will not accumulate on weekends or holidays recognized by the DOE-AL facility.

- 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. Unusable data resulting from non-compliance will not be paid for.
- e) **The DOE-AL AMP guidelines for reduction in payment are provided as Attachment XX.**

#### 4.2.4 Reporting PE results

The reports for PE samples submitted by the DOE-AL facility SMO shall be due 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 quality control 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 DOE-AL facility.

## 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 site-specific plan for DOE-AL facility samples, 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, ILMO4.0.

U.S. Environmental Protection Agency, Contract Laboratory Program Statement of Work for Organics Analysis, Multi-media, Multi-Concentration, OLMO3.2, 1994.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Report SW-846, Third Edition, November 1986 (and updates, including Proposed Update III).

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.

## 7.0 ATTACHMENTS

**(Insert and number the attachments listed in bold type.)**

- Attachment 1            General Inorganic parameters and MDLs.
- Attachment 2            Radiochemical parameters and MDCs.
- Attachment 3            Organic parameters and MDLs.
- Attachment 4            Geotechnical methods.
- Attachment 5            Holding times and preservation techniques.
- Attachment 6            Target surrogate recovery control limits.
- **Attachment X.X        COC/analysis request form.**
- **Attachment X.X        Format of EDD.**
- **Attachment X.X        Parameter codes.**
- **Attachment X.X        DOE-AL AMP information sharing policy.**
- **Attachment X.X        DOE-AL AMP payment reduction policy.**



## GLOSSARY

ASTM Type I, II	For the purposes of this SOW, ASTM Type I water is water having conductivity less than 0.06 $\mu\text{mho/cm}$ or resistivity greater than 16.67 $\text{M}\Omega\text{-cm}$ . ASTM Type II water is water having conductivity less than 1 $\mu\text{mho/cm}$ or resistivity greater than 1 $\text{M}\Omega\text{-cm}$ . The applicable ASTM standard is D 1193-77.
Chemical Analysis	The term chemical analysis 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	Controlled documents are those 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	Daily requirements for checking refrigerators, balances, and the like apply only to business days. Daily requirements for instrument calibration and standards preparation refer only to days when the instruments are used.
Deliverable Levels	The deliverable levels are specifications for classes of analytical data reports.
Delivery Order	A delivery order is a specific request for analysis of a sample or samples under an existing contract that provides all applicable specifications. This means that no technical specifications are included with a delivery order except when special conditions occur.
DLC	DLC is the acronym for decision level concentration. When calculated according to the equation in this SOW, the DLC gives the level at which there is a five percent probability of reporting a false positive for a sample containing no analyte. The DLC is calculated sample specifically using variable values from the actual analytical conditions.
Duplicate	A duplicate is a sample split taken by the sampling team and submitted as a sample for the purpose of assessing both sampling and analytical precision.
EDD	EDD is the acronym for electronic data deliverable. This is the computer file containing analytical results and associated information.
Intermediate Dilution	An intermediate dilution is a dilution of some stock solution that requires further dilution before use in instrument calibration or quality control sample preparation. Intermediate dilutions are not used to calibrate instruments in undiluted form.
MDC	MDC is the acronym for minimum detectable concentration. The MDC provides general information about the sensitivity of analytical techniques in radiochemistry based upon assumed nominal conditions.
MDL	MDL is the acronym for method detection limit. This is a measure of instrument sensitivity using solutions that have been subjected to all sample preparation

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steps for the method. Reagent contributions to the signal are thus included in the MDL. MDLs are determined only for organic parameters under this SOW.

Optional Compounds	Optional compounds are those that are not routinely required in instrument calibration or the reporting of analytical results.
PE Samples	Performance evaluation (PE) samples are 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	PQL is the acronym for practical quantitation limit. The PQL is defined to be five times the MDL under this SOW.
Reagent	Reagents are chemicals of known purity that are used in analytical methods. This term does not apply to materials used to calibrate instruments or to perform quality control activities. Such materials are called standards.
Record	Record is the term applied to information that is subject to special handling requirements. In this SOW, the term 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 replicate is a sample split taken by the laboratory and prepared separately from the original sample for the purpose of assessing analytical precision.
SMO	The sample management office is the entity within the DOE-AL facility that is responsible for writing technical and quality assurance specifications for analytical chemistry, technical administration of laboratory contracts, sample shipment and tracking, and the various data verification, validation, and management functions.
SOP	SOP is 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	A standard is any material intended for use, possibly as a dilution, in instrument calibration or to perform quality control activities.
Stock Solution	A stock solution is a high-concentration standard. Stock solutions are not used to calibrate instruments or as quality control samples, but rather are diluted to produce the standards used to calibrate or prepare quality control samples.
Turn Around Time	Turn around time refers to the period between receipt of samples by the laboratory and receipt of the analytical report for those samples by the DOE-AL facility SMO.
Working Standards	Working standards are those used to calibrate instruments.

## GLOSSARY

Worksheet	The term worksheet 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	Worksheet review is a process for assessing the degree of compliance with laboratory and client requirements in the analysis documentation.

## GLOSSARY

**Table I.** Metal Target Analytes and Required MDLs.

Analyte	MDL		
	Water µg/L	Solid <sup>1</sup> µg/kg	Air Filter µg/sample
Aluminum	45	2250	10
Antimony	3	150	1
Arsenic	1	50	2
Barium	2	100	10
Beryllium	0.3	15	1
Boron	5	250	NA
Cadmium	0.1	5	1
Calcium	10	500	NA
Chromium	7	350	5
Cobalt	7	350	1
Copper	6	300	1
Iron	7	350	10
Lithium			NA
Lead	1	50	2
Magnesium	30	1500	10
Manganese	2	100	5
Mercury	0.2	20	0.005
Molybdenum	8	400	1
Nickel	15	750	1
Potassium		250	10
Selenium	2	100	2
Silver	7	350	1
Silica	58	2900	NA
Sodium	29	1450	100
Strontium			
Thallium	1	50	1
Tin			
Titanium			NA
Uranium	0.1	5	1
Vanadium	8	400	1
Zinc	2	100	2

<sup>1</sup>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.

## GLOSSARY

**Table II.** Miscellaneous General Inorganic Target Analytes, Methods, and Required MDLs.

Analyte	Method Nos.	MDL	
		Water mg/L	Solid mg/kg
Acidity as CaCO <sub>3</sub>	305	10	NA
Alkalinity as CaCO <sub>3</sub>	310	10	NA
Ammonium as N	350	0.01	0.2
Bicarbonate/carbonate	SM 2320B	10	NA
Biological oxygen demand (BOD), 5 day	405	2	NA
Bromide	300	0.01	0.2
Carbon, dissolved organic (DOC)	415	1	NA
Carbon, total organic (TOC)	415, 9060	1	100
Chemical oxygen demand (COD)	410	5	NA
Chloride	300, 325, 9250	0.02	0.4
Chromium (VI)	7196A	0.5	2.5
Color (color units)	110	1	NA
Corrosivity (mm/year)	1110	NA	NA
Cyanide, total	9010B, 9014	0.02	0.4
Dissolved oxygen (DO)	360	0.05	NA
Fluoride	300.0, 340	0.01	0.2
Hardness as CaCO <sub>3</sub>	130	10	NA
Ignitability (°C)	1010, 1020A	1	1
Iodide	300		
Nitrate as N	300, 353, 9200	0.002	0.04
Nitrate + nitrite as N	300, 353	0.002	0.04
Nitrite as N	300, 253	0.004	0.08
Oil and grease	1664, 9070	5	100
Perchlorate	314, 9058, LC/MS/MS	1	
pH (unitless)	150, 9040B, 9045C	0.1	0.1
Phenols, total recoverable	9065, 9066	0.05	1
o-Phosphate as P	300	0.003	0.06
Phosphorus, total as P	365	0.01	0.2
Sulfide	376	1	20
Sulfate	300	0.02	0.4
Specific conductance (µmho/cm)	120, 9050	1	NA
Solids, settleable (mL/L/hr.)	160	0.2	NA
Solids, total (TS)	160	10	NA
Solids, total dissolved (TDS)	160	10	NA
Solids, total suspended (TSS)	160	3	NA
Solids, volatile	160	20	NA
Total organic halide (TOX)	9020B	0.005	NA
Total Kjeldahl nitrogen	351	0.05	1
Total petroleum hydrocarbons	418, 9073	1	20
Turbidity (NTU)	180	0.05	NA
Uranium, total	908	0.001	0.02

## GLOSSARY

**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	0.136
α-BHC	319-84-6	0.003	0.102
β-BHC	319-85-7	0.006	0.204
δ-BHC	319-86-8	0.009	0.306
γ-BHC (Lindane)	58-89-9	0.004	0.136
α-Chlordane	5103-71-9		
γ-Chlordane	5103-74-2		
4,4'-DDD	72-54-8	0.012	0.404
4,4'-DDE	72-55-9	0.012	0.408
4,4'-DDT	50-29-3	0.012	0.408
Dieldrin	60-57-1	0.002	0.068
Endosulfan I	959-98-8	0.014	0.476
Endosulfan II	33213-65-9	0.004	0.136
Endosulfan sulfate	1031-07-8	0.066	2.244
Endrin	72-20-8	0.006	0.204
Endrin aldehyde	7421-93-4	0.023	0.782
Endrin ketone	53494-70-5		
Heptachlor	76-44-8		
Heptachlor epoxide	1024-57-3	0.083	2.822
4,4'-Methoxychlor	72-43-5		
Toxaphene	8001-35-2	0.24	8.16

## GLOSSARY

**Table III.** Polychlorinated Biphenyls (PCBs) Target Analyte List and MDLs.

Compound Name	CAS No.	Water MDL, $\mu\text{g/L}$	Solid MDL, $\mu\text{g/kg}$
Aroclor - 1016	12674-11-2	0.065	2.145
Aroclor - 1221	11104-28-2		
Aroclor - 1232	11141-16-5		
Aroclor - 1242	53469-21-9		
Aroclor - 1248	12672-29-6		
Aroclor - 1254	11097-69-1		
Aroclor - 1260	11096-82-5		

**Table IV.** Chlorinated Herbicides Target Analyte List and MDLs.

Compound Name	CAS No.	Water MDL, $\mu\text{g/L}$	Solid MDL, $\mu\text{g/kg}$
Acifluorfen	50594-66-6	0.096	3.168
Betazon	25057-89-0	0.2	6.6
Chloramben	133-90-4	0.093	3.069
2,4-D	94-75-7	0.2	6.6
2,4-DB	94-82-6	0.8	26.4
Dalapon	75-99-0	1.3	42.9
Dicamba	1918-00-9	0.081	2.673
3,5-Dichlorobenzonic acid	51-36-5	0.061	2.013
Dichlorprop	120-36-5	0.26	8.58
Dinoseb	88-85-7	0.19	6.27
MCPA	94-74-6		
MCPP	93-65-2		
4-Nitrophenol	100-02-1	0.13	4.29
Pentachlorophenol	87-86-5	0.076	2.508
Picloram	1918-02-1	0.14	4.62
2,4,5-T	93-76-5	0.08	2.64
2,4,5-TP (Silvex)	93-72-1	0.075	2.475

## GLOSSARY

**Table V.** VOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Low-Level Solid Samples.

Compound Name	CAS No.	25 ml purge MDL $\mu\text{g/L}$	5 gm purge MDL $\mu\text{g/kg}$
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	0.04	0.2
Bromobenzene	108-86-1	0.03	0.15
Bromochloromethane	74-97-5	0.04	0.2
Bromodichloromethane	75-27-4	0.08	0.4
Bromoform	75-25-2	0.12	0.6
Bromomethane	74-83-9	0.11	0.55
2-Butanone	78-93-3		
n-Butylbenzene	104-51-8	0.11	0.55
sec-Butylbenzene	135-98-8	0.13	0.65
tert-Butylbenzene	98-06-6	0.14	0.7
Carbon disulfide	75-15-0		
Carbon tetrachloride	56-23-5	0.21	1.05
Chloral hydrate	75-87-6		
Chlorobenzene	108-90-7	0.04	0.2
Chloroethane	75-00-3	0.10	0.5
Chloroform	67-66-3	0.03	0.15
Chloromethane	74-87-3	0.13	0.65
Chloroprene	126-99-8		
2-Chlorotoluene	95-49-8	0.04	0.2
4-Chlorotoluene	106-43-4	0.06	0.3

## GLOSSARY

**Table V. (continued)** VOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Low-Level Solid Samples.

Compound Name	CAS No.	25 ml purge MDL $\mu\text{g/L}$	5 gm purge MDL $\mu\text{g/kg}$
Dibromochloromethane	124-48-1	0.05	0.25
1,2-Dibromo-3-chloropropane	96-12-8	0.26	1.3
1,2-Dibromoethane	106-93-4	0.06	0.3
Dibromomethane	74-95-3	0.24	1.2
1,2-Dichlorobenzene	95-50-1	0.03	0.15
1,3-Dichlorobenzene	541-73-1	0.12	0.6
1,4-Dichlorobenzene	106-46-7	0.03	0.15
Dichlorodifluoromethane	75-71-8	0.10	0.5
1,1-Dichloroethane	75-34-3	0.04	0.2
1,2-Dichloroethane	107-06-2	0.06	0.3
1,1-Dichloroethene	75-35-4	0.12	0.6
cis-1,2-Dichloroethene	156-59-2	0.12	0.6
trans -1,2-Dichloroethene	156-60-5	0.06	0.3
1,2-Dichloroethene (total)	540-59-0		
1,2-Dichloropropane	78-87-5	0.04	0.2
1,3-Dichloropropane	142-28-9	0.04	0.2
2,2-Dichloropropane	594-20-7	0.35	1.75
1,1-Dichloropropene	563-58-6	0.10	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	0.06	0.3
Ethyl methacrylate	97-63-2		
Hexachlorobutadiene	87-68-3	0.11	0.55
2-Hexanone	591-78-6		

## GLOSSARY

**Table V. (continued)** VOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Low-Level Solid Samples.

Compound Name	CAS No.	25 ml purge MDL $\mu\text{g/L}$	5 gm purge MDL $\mu\text{g/kg}$
Iodomethane	74-88-4		
Isobutyl alcohol	78-83-1		
Methacrylonitrile	126-98-7		
Methyl methacrylate	80-62-6		
Isopropylbenzene	98-82-8	0.15	0.75
4-Isopropyltoluene	99-87-6	0.12	0.6
Methylene chloride	75-09-2	0.03	0.15
4-Methyl-2-pentanone	108-10-1		
Naphthalene	91-20-3	0.04	0.2
2-Picoline	109-06-8		
Propionitrile	107-12-0		
n-Propylbenzene	103-65-1	0.04	0.2
Styrene	100-42-5	0.04	0.2
1,1,1,2-Tetrachloroethane	630-20-6	0.05	0.25
1,1,2,2-Tetrachloroethane	79-34-5	0.04	0.2
Tetrachloroethene	127-18-4	0.14	0.7
Toluene	108-88-3	0.11	0.55
1,2,3-Trichlorobenzene	87-61-6	0.03	0.15
1,2,4-Trichlorobenzene	120-82-1	0.04	0.2
1,1,1-Trichloroethane	71-55-6	0.08	0.4
1,1,2-Trichloroethane	79-00-5	0.10	0.5
Trichloroethene	79-01-6	0.19	0.95
Trichlorofluoromethane	75-69-4	0.08	0.4
1,2,3-Trichloropropane	96-18-4	0.32	1.60
1,2,4-Trimethylbenzene	95-63-6	0.13	0.65
1,3,5-Trimethylbenzene	108-67-8	0.13	0.65
Vinyl acetate	108-05-4		
Vinyl chloride	75-01-4	0.17	0.85
o-Xylene	95-47-6	0.11	0.55
m-Xylene	108-38-3	0.05	0.25
p-Xylene	106-42-3	0.13	0.65
Xylenes (total)	1330-20-7		

## GLOSSARY

**Table VI.** SVOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Solid Samples.

Compound Name	CAS No.	MDLs <sup>1</sup>	
		Water $\mu\text{g/L}$	Solid $\mu\text{g/kg}$
Acenaphthene	83-32-9	1.9	62.7
Acenaphthylene	208-96-8	3.5	115.5
Acetophenone	98-86-2		
2-Acetylaminofluorene	53-96-3		
Aldrin	309-00-2	1.9	62.7
4-Aminobiphenyl	92-67-1		
Aniline	62-53-3		
Anthracene	120-12-7	1.9	62.7
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	36	1188
Aroclor - 1260	11096-82-5		
Azobenzene	103-33-3		
Benzidine	92-87-5	44	1452
Benz(a)anthracene	56-55-3	7.8	257.4
Benzo(b)fluoranthene	205-99-2	4.8	158.4
Benzo(k)fluoranthene	207-08-9	2.5	82.5
Benzo(g,h,i)perylene	191-24-2	4.1	1455
Benzo(a)pyrene	50-32-8	2.5	825
Benzoic acid	65-85-0		
Benzyl alcohol	100-51-6		
$\alpha$ -BHC	319-84-6		
$\beta$ -BHC	319-85-7	4.2	138.6

## GLOSSARY

**Table VI. (continued)** SVOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Solid Samples.

Compound Name	CAS No.	MDLs <sup>1</sup>	
		Water $\mu\text{g/L}$	Solid $\mu\text{g/kg}$
$\delta$ -BHC	319-86-8	3.1	102.3
$\gamma$ -BHC (Lindane)	58-89-9		
Bis(2-chloroethoxy) methane	111-91-1	5.3	174.9
Bis(2-chloroethyl) ether	111-44-4	5.7	188.1
Bis(2-chloroisopropyl) ether	108-60-1	5.7	188.1
Bis(2-ethylhexyl) phthalate	117-81-7	2.5	82.5
4-Bromophenyl phenyl ether	101-55-3	1.9	62.7
Butyl benzyl phthalate	85-68-7	2.5	82.5
Carbazole	86-74-8		
Chlordane	57-74-9		
4-Chloroaniline	106-47-8		
Chlorobenzilate	510-15-6		
4-Chloro-3-methylphenol	59-50-7	3.0	99
2-Chloronaphthalene	91-58-7	1.9	62.7
2-Chlorophenol	95-57-8	3.3	108.9
4-Chlorophenyl phenyl ether	7005-72- 3	4.2	138.6
Chrysene	218-01-9	2.5	82.5
4,4'-DDD	72-54-8	2.8	92.4
4,4'-DDE	72-55-9	5.6	184.8
4,4'-DDT	50-29-3	4.7	155.1
Demeton-O	298-03-3		
Demeton-S	126-75-0		
Diallate (cis or trans)	2303-16- 4		
Dibenz(a,h)anthracene	53-70-3	2.5	82.5
Dibenzofuran	132-64-9		

## GLOSSARY

**Table VI. (continued)** SVOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Solid Samples.

Compound Name	CAS No.	MDLs <sup>1</sup>	
		Water $\mu\text{g/L}$	Solid $\mu\text{g/kg}$
Di-n-butyl phthalate	84-74-2		
1,2-Dichlorobenzene	95-50-1	1.9	62.7
1,3-Dichlorobenzene	541-73-1	1.9	62.7
1,4-Dichlorobenzene	106-46-7	4.4	145.2
3,3'-Dichlorobenzidine	91-94-1	16.5	544.5
2,4-Dichlorophenol	120-83-2	2.7	89.1
2,6-Dichlorophenol	87-65-0		
Dieldrin	60-57-1	2.5	82.5
Diethyl phthalate	84-66-2	1.9	62.7
Dimethoate	60-51-5		
p-	60-11-7		
Dimethylaminoazobenzene			
7,12-	57-97-6		
Dimethylbenz(a)anthracene			
3,3'-Dimethylbenzidine	119-93-7		
2, 4-Dimethylphenol	105-67-9	2.7	89.1
Dimethyl phthalate	131-11-3	1.6	52.8
1,3-Dinitrobenzene	99-65-0		
4,6-Dinitro-2-methylphenol	534-52-1	24	792
2,4-Dinitrophenol	51-28-5	42	1386
2,4-Dinitrotoluene	121-14-2	5.7	188.1
2,6-Dinitrotoluene	606-20-2	1.9	62.7
Dinoseb	88-85-7		
Diphenylamine	122-39-4		
Di-n-octyl phthalate	117-84-0	2.5	82.5
Disulfoton	298-04-4		

## GLOSSARY

**Table VI. (continued)** SVOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Solid Samples.

Compound Name	CAS No.	MDLs <sup>1</sup>	
		Water $\mu\text{g/L}$	Solid $\mu\text{g/kg}$
Endosulfan I	959-98-8		
Endosulfan II	33213-65-9		
Endosulfan sulfate	1031-07-8	5.6	184.8
Endrin	72-20-8		
Endrin aldehyde	7421-93-4		
Ethyl methanesulfonate	62-50-0		
Famphur	52-85-7		
Fluoranthene	206-44-0	2.2	72.6
Fluorene	86-73-7	1.9	62.7
Heptachlor	76-44-8	1.9	62.7
Heptachlor epoxide	1024-57-3	2.2	72.6
Hexachlorobenzene	118-74-1	1.9	62.7
Hexachlorobutadiene	87-68-3	0.9	29.7
Hexachlorocyclopentadiene	77-47-4		
Hexachloroethane	67-72-1	1.6	52.8
Hexachlorophene	70-30-4		
Hexachloropropene	1888-71-7		
Indeno(1,2,3-cd)pyrene	193-39-5	3.7	122.1
Isodrin	465-73-6		
Isophorone	78-59-1	2.2	72.6
Isosafrole	120-58-1		
Kepone	143-50-0		
Methapyrilene	91-80-5		
Methoxychlor	72-43-5		

## GLOSSARY

**Table VI. (continued)** SVOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Solid Samples.

Compound Name	CAS No.	MDLs <sup>1</sup>	
		Water $\mu\text{g/L}$	Solid $\mu\text{g/kg}$
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	1.6	52.8
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	1.9	62.7
2-Nitrophenol	88-75-5	3.6	118.8
4-Nitrophenol	100-02-7	2.4	79.2
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	1.9	62.7
N-Nitroso-di-n-propylamine	621-64-7		
N-Nitrosomethylethylamine	10595-95-6		

## GLOSSARY

**Table VI. (continued)** SVOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Solid Samples.

Compound Name	CAS No.	MDLs <sup>1</sup>	
		Water $\mu\text{g/L}$	Solid $\mu\text{g/kg}$
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	3.6	118.8
Phenacetin	62-44-2		
Phenanthrene	85-01-8	5.4	178.2
Phenol	108-95-2	1.5	49.5
1,4-Phenylenediamine	106-50-3		
Phorate	298-02-2		
2-Picoline	109-06-8		
Pronamide	23950-58-5		
Pyrene	129-00-0	1.9	62.7
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		

## GLOSSARY

**Table VI. (continued)** SVOC Method Target Analyte List and MDLs in  $\mu\text{g/L}$  for Water Samples and  $\mu\text{g/kg}$  for Solid Samples.

Compound Name	CAS No.	MDLs <sup>1</sup>	
		Water $\mu\text{g/L}$	Solid $\mu\text{g/kg}$
o-Toluidine	95-53-4		
Toxaphene	8001-35-2		
1,2,4-Trichlorobenzene	120-82-1	1.9	62.7
2,4,5-Trichlorophenol	95-95-4		
2,4,6-Trichlorophenol	88-06-2	2.7	89.1
0,0,0-Triethyl phosphorothioate	126-68-1		

## GLOSSARY

**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		

## GLOSSARY

**Table VIII.** 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	0.035	13.48
4-Amino-2,6-Dinitrotoluene (4-Am-DNT)	1946-51-0	0.060	23.1
1,3-Dinitrobenzene (DNB)	99-65-0	0.11	42.35
2,4-Dinitrotoluene (24DNT)	121-14-2	0.02	7.7
2,6-Dinitrotoluene (26DNT)	606-20-2	0.31	119.4
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	0.84	323.4
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8		
Nitrobenzene (NB)	98-95-3		
Nitroglycerine (NG)			
2-Nitrotoluene (2NT)	88-72-2		
3-Nitrotoluene (3NT)	99-08-1		
4-Nitrotoluene (4NT)	99-99-0		
Pentaerythritol tetranitrate (PETN)			
Octahydro-1,3,5,7-tetranitro-1,3,5,7- tetrazocine (HMX)	2691-41-0		
1,3,5-Trinitrobenzene (135TNB)	99-35-4	0.26	100.1
2,4,6-Trinitrotoluene (TNT)	118-96-7	0.11	42.35

## GLOSSARY

**Table IX.** 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

## GLOSSARY

**Table I.** Required Gamma Spectroscopy Radionuclides and Minimum Detectable Concentrations (MDC) by Matrix. (July 2000)

Radionuclide	MDC				
	Water pCi/L	Solid pCi/g	Air Filter pCi/sample	Urine pCi/L	Vegetation pCi/g
Ann. Rad.	TBD	TBD	TBD	TBD	TBD
<sup>7</sup> Be	TBD	TBD	TBD	TBD	TBD
<sup>22</sup> Na	TBD	TBD	TBD	TBD	TBD
<sup>40</sup> K	TBD	TBD	TBD	TBD	TBD
<sup>51</sup> Cr	TBD	TBD	TBD	TBD	TBD
<sup>54</sup> Mn	TBD	TBD	TBD	TBD	TBD
<sup>57</sup> Co	TBD	TBD	TBD	TBD	TBD
<sup>59</sup> Fe	TBD	TBD	TBD	TBD	TBD
<sup>60</sup> Co	10	0.1	20	200	0.5
<sup>65</sup> Zn	TBD	TBD	TBD	TBD	TBD
<sup>75</sup> Se	TBD	TBD	TBD	TBD	TBD
<sup>85</sup> Sr	TBD	TBD	TBD	TBD	TBD
<sup>88</sup> Y	TBD	TBD	TBD	TBD	TBD
<sup>95</sup> Nb	TBD	TBD	TBD	TBD	TBD
<sup>95</sup> Zr	TBD	TBD	TBD	TBD	TBD
<sup>103</sup> Ru	TBD	TBD	TBD	TBD	TBD
<sup>106</sup> Rh	TBD	TBD	TBD	TBD	TBD
<sup>106</sup> Ru	TBD	TBD	TBD	TBD	TBD
<sup>109</sup> Cd	TBD	TBD	TBD	TBD	TBD
<sup>113</sup> Sn	TBD	TBD	TBD	TBD	TBD
<sup>124</sup> Sb	TBD	TBD	TBD	TBD	TBD
<sup>125</sup> Sb	TBD	TBD	TBD	TBD	TBD
<sup>133</sup> Ba	TBD	TBD	TBD	TBD	TBD
<sup>133</sup> I	TBD	TBD	TBD	TBD	TBD
<sup>134</sup> Cs	TBD	TBD	TBD	TBD	TBD
<sup>137</sup> Cs	10	0.1	20	200	0.5
<sup>139</sup> Ce	TBD	TBD	TBD	TBD	TBD
<sup>141</sup> Ce	TBD	TBD	TBD	TBD	TBD
<sup>144</sup> Ce	TBD	TBD	TBD	TBD	TBD
<sup>152</sup> Eu	TBD	TBD	TBD	TBD	TBD
<sup>154</sup> Eu	TBD	TBD	TBD	TBD	TBD
<sup>203</sup> Hg	TBD	TBD	TBD	TBD	TBD
<sup>208</sup> Tl	TBD	TBD	TBD	TBD	TBD
<sup>211</sup> Bi	TBD	TBD	TBD	TBD	TBD
<sup>211</sup> Pb	TBD	TBD	TBD	TBD	TBD
<sup>212</sup> Bi	TBD	TBD	TBD	TBD	TBD
<sup>212</sup> Pb	TBD	TBD	TBD	TBD	TBD
<sup>214</sup> Bi	TBD	TBD	TBD	TBD	TBD
<sup>214</sup> Pb	TBD	TBD	TBD	TBD	TBD
<sup>219</sup> Rn	TBD	TBD	TBD	TBD	TBD

TBD: Minimum detectable concentration is to be determined by the subcontract laboratory.

## GLOSSARY

**Table I. (continued)** Required Gamma Spectroscopy Radionuclides and Minimum Detectable Concentrations (MDC) by Matrix.

Radionuclide	MDC				
	Water pCi/L	Solid pCi/g	Air Filter pCi/sample	Urine pCi/L	Vegetation pCi/g
<sup>223</sup> Ra	TBD	TBD	TBD	TBD	TBD
<sup>224</sup> Ra	TBD	TBD	TBD	TBD	TBD
<sup>226</sup> Ra	TBD	TBD	TBD	TBD	TBD
<sup>227</sup> Th	TBD	TBD	TBD	TBD	TBD
<sup>228</sup> Ac	TBD	TBD	TBD	TBD	TBD
<sup>228</sup> Ra	TBD	TBD	TBD	TBD	TBD
<sup>231</sup> Pa	TBD	TBD	TBD	TBD	TBD
<sup>231</sup> Th	TBD	TBD	TBD	TBD	TBD
<sup>233</sup> Pa	TBD	TBD	TBD	TBD	TBD
<sup>234m</sup> Pa	TBD	TBD	TBD	TBD	TBD
<sup>234</sup> Th	TBD	TBD	TBD	TBD	TBD
<sup>235</sup> U	TBD	TBD	TBD	TBD	TBD
<sup>237</sup> Np	TBD	TBD	TBD	TBD	TBD
<sup>238</sup> U	TBD	TBD	TBD	TBD	TBD
<sup>239</sup> Np	TBD	TBD	TBD	TBD	TBD
<sup>241</sup> Am	TBD	TBD	TBD	TBD	TBD

TBD: Minimum detectable concentration is to be determined by the subcontract laboratory.

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**Table II.** Alpha Spectrometry Radionuclides and Required Minimum Detectable Concentrations (MDC) by Matrix.

Radionuclide	MDC					
	Water pCi/L	Solid pCi/g	Air Filter pCi/sample	Urine pCi/L	Vegetation pCi/g	Feces Ash pCi/g
<sup>241</sup> Am	0.1	0.1	0.1	0.1	0.01	0.04
<sup>244</sup> Cm	0.1	0.1	0.1	0.1	0.01	0.04
<sup>237</sup> Np	0.1	0.1	0.1	0.1	0.01	0.04
<sup>210</sup> Po	1	0.5	1	1	0.5	0.5
<sup>238</sup> Pu	0.1	0.1	0.1	0.1	0.01	0.04
<sup>239</sup> Pu	0.1	0.1	0.1	0.1	0.01	0.04
<sup>226</sup> Ra	1	0.5	1	1	0.5	0.5
<sup>228</sup> Th	0.1	0.1	0.1	0.1	0.1	0.1
<sup>230</sup> Th	0.1	0.1	0.1	0.1	0.1	0.1
<sup>232</sup> Th	0.1	0.1	0.1	0.1	0.1	0.1
<sup>234</sup> U	0.1	0.1	0.1	0.1	0.01	0.1
<sup>235</sup> U	0.1	0.1	0.1	0.1	0.01	0.1
<sup>238</sup> U	0.1	0.1	0.1	0.1	0.01	0.1

**Table III.** Gas Proportional Counting Radionuclides and Required Minimum Detectable Concentrations (MDC) by Matrix.

Radionuclide	MDC			
	Water pCi/L	Solid pCi/g	Air Filter pCi/sample	Vegetation pCi/g
Gross $\alpha$		1		1
Gross $\beta$		1		1
<sup>89</sup> Sr		1	5	2
<sup>90</sup> Sr		1	5	2
<sup>131</sup> I		2	5	5
<sup>210</sup> Pb		1	5	2
<sup>210</sup> Po		1	1	2
<sup>226</sup> Ra		1	1	2
<sup>228</sup> Ra		0.5	0.5	1
<sup>99</sup> Tc		1	5	10

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**Table IV.** Liquid Scintillation Counting Radionuclides and Required Minimum Detectable Concentrations (MDC) by Matrix.

Radionuclide	MDC			
	Water pCi/L	Solid pCi/g	Air Filter pCi/sample	Swipe pCi/100cm <sup>2</sup>
<sup>3</sup> H	250	250 <sup>1</sup> (pCi/L)	10	10
<sup>14</sup> C	500	10	20	20
<sup>99</sup> Tc	5	10	20	20
<sup>210</sup> Pb	1.0	5	10	10
<sup>222</sup> Rn	200	200		

<sup>1</sup>For tritium the specified solid MDC applies to the extracted water.

## GLOSSARY

**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).

## GLOSSARY

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#### 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 8260B: 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 8270C: 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%

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#### Method 8330: 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-Dinitrobenzene (recommended)	100-25-4	50-160%	50-160%
1,2-Dinitrobenzene (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%

## GLOSSARY

305.1,310	Acidity, Alkalinity	Water	500 mL Plastic or Glass	4 °C	1
300.0, 320.1, 325, 340, 375	Bromide, Chloride Fluoride, Sulfate	Water	1 L Plastic	4 °C	2
405.1	BOD	Water	1 L Plastic	4 °C	4
9010B, 9013, 9014, 335.1, 335.3	Total Cyanide Amenable Cyanide	Water Solid/Other	1 L Plastic 125 mL Glass Jar	4 °C; NaOH; pH > 12 4 °C	1 1
415, 9060	DOC, TOC	Water Solid/Other	250 mL Amber Glass 125 mL Glass Jar	4 °C; H <sub>2</sub> SO <sub>4</sub> ; pH < 2 4 °C	2 2
200.7, 200.8, 6010B, 6020,	All metals except Cr(VI) and Hg	Water Solid/Other	500 mL Plastic 250 mL Glass Jar	HNO <sub>3</sub> ; pH < 2	1 1
3060A, 7196A, 7197	Cr(VI)	Water Solid/Other	500 mL Plastic 250 mL Glass Jar	4 °C 4 °C	2 3
245.1, 7470A, 7471A	Hg	Water Solid/Other	500 mL Plastic 250 mL Glass Jar	HNO <sub>3</sub> ; pH < 2 4 °C	2 2
130.1	Hardness	Water		HNO <sub>3</sub> ; pH < 2 4 °C	1
345.1	Iodide	Water	500 mL Plastic or Glass	4 °C	2
353, 351, 365.4, 350	Ammonium, Nitrate + Nitrite Total Phosphorus, TKN	Water	1 L Plastic	4 °C; H <sub>2</sub> SO <sub>4</sub> ; pH < 2	2
300.0 354.1	Nitrate, Nitrite, Ortho Phosphorus	Water	500 mL Plastic	4 °C	4
365	Ortho Phosphorus	Water	500 mL Plastic	4 °C; H <sub>2</sub> SO <sub>4</sub> ; pH < 2	4
9210/9211	Nitrate	Water Solid/Other	1 L Plastic 250 mL Glass Jar	4 °C; 1M Boric Acid 4 °C	4 4
314.0, 9058	Perchlorate	Water	250 mL Plastic or Glass	4 °C	2
6321 (modified)	Perchlorate by LC/MS/MS	Water Solid	250 mL Plastic or Glass 4 oz. Wide-mouth jar	4 °C 4 °C	2 2
410	Chemical Oxygen Demand (COD)	Water	250 mL Glass	4 °C; H <sub>2</sub> SO <sub>4</sub> ; pH < 2	2
1664 2	Total Recoverable oil and Grease 28 Days	Water NA Solid/Other	1 L Glass  125 mL Glass Jar	4 °C; H <sub>2</sub> SO <sub>4</sub> or HCl; pH <  4 °C	  2
9070/9071A	Total Recoverable Oil and Grease	Water Solid/Other	1 L Glass 125 mL Glass Jar	4 °C; HCl; pH < 2 4 °C	2 2
ASTM D-854	Specific Gravity				

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376/9030B/9031	Sulfide acetate; pH > 9	Water	1 L Glass	4 °C; NaOH; Zinc	
	7 Days	NA			
		Solid/Other	125 mL Glass Jar	4 °C	7
160	TDS, TSS, TS	Water	1 L Plastic	4 °C	7
160.4	volatile solids (volatile residue)	Water	plastic or glass	4 °C	7
9020B	TOX	Water	1 L Amber Glass	4 °C; H <sub>2</sub> SO <sub>4</sub> ; pH < 2	2
		Solid/Other	125 mL Glass Jar	4 °C	2
9060	TOC	Water	Glass	4 °C; H <sub>2</sub> SO <sub>4</sub> or HCL; pH	
< 2	2 hours, unless				
after collection	acidified	N/A		if analyzed >2 hours	
418.1	TPH	Water	1 L Amber Glass	4 °C; HCl; pH < 2	2
1664	TPH	Water	1 L Amber Glass	4 °C; H <sub>2</sub> SO <sub>4</sub> or HCl; pH <	
2	28 Days	NA			
8440	TPH	Solid/Other	125 mL Glass Jar	4 °C	2
9065, 9066	Total Recoverable Phenols	Water	1 L Glass	4 °C; H <sub>2</sub> SO <sub>4</sub> ; pH < 4	2
420		Solid	125 mL Glass Jar	4 °C	2
150.1, 9040B	pH	Water	125 mL Plastic	4 °C	2
110, 180.1	Color, Turbidity	Water	500 mL Plastic	4 °C	4
120.1, 9050	Specific Conductance	Water	125 mL Plastic	4 °C	2
All radiochemical parameters except Rn-222 and tritium		Water	1 L Plastic (2 x 2 L Preferred)	HNO <sub>3</sub> ; pH < 2	1
		Solid/Other	250 mL Glass Jar		1
913.0	Radon 222	Water	125 mL Glass	None	7
906.0	Tritium	Water	1 L Glass		1
		Solid/Other	Sample size will vary with moisture content		1
8015 (Modified)	Petroleum Hydrocarbons (Diesel Range Organics)	Water	2 x 1 L Amber Glass Bottle	4 °C	7
		Soil/Other	250 mL Glass Jar	4 °C	1
	Petroleum Hydrocarbons (Gasoline Range Organics)	Water	3 x 40 mL Glass Vial	4 °C; HCl; pH < 2	1
		Soil/Other	125 mL Glass Jar	4 °C	1
5035/8015 (Modified) Preservative	Petroleum Hydrocarbons (Gasoline Range Organics)	Soil	4 x 40 mL Glass Vial	4 °C, 2 Vials NaHSO <sub>4</sub> , 1 Vial CH <sub>3</sub> OH, 1 Vial No	*
8021B	Halogenated Volatile Organics	Water	3 x 40 mL Glass Vial	4 °C; HCl; pH < 2	1
		Soil/Other	125 mL Glass Jar	4 °C	1
5035/8021B Preservative	Halogenated Volatile Organics	Soil	4 x 40 mL Glass Vial	4 °C, 2 Vials NaHSO <sub>4</sub> , 1 Vial CH <sub>3</sub> OH, 1 Vial No	*
8081, 8082	Organochlorine Pesticides, PCBs	Water	4 L Amber Glass Bottle	4 °C	7
		Soil/Other	250 Glass Jar	4 °C	1

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8141A pH 5-8	Organophosphorous Compounds 7 Days	Water 40 Days Soil/Other	4 L Amber Glass Bottle 250 Glass Jar	4 °C; NaOH or H <sub>2</sub> SO <sub>4</sub> ; 4 °C	1
8151A	Chlorinated Herbicides	Water Soil/Other	4 L Amber Glass Bottle 250 Glass Jar	4 °C; 4 °C	7 1
8260B (Modified)	Volatile Organics by GC-MS	Water Soil/Other	3 x 40 mL Glass Vial 125 mL Glass Jar	4 °C; HCl; pH < 2 4 °C	1 1
5035/8260B Preservative	Volatile Organics by GC-MS	Soil	4 x 40 mL Glass Vial	4 °C, 2 Vials NaHSO <sub>4</sub> , 1 Vial CH <sub>3</sub> OH, 1 Vial No	*
8270C	Semi-volatile Organics by GC-MS	Water Soil/Other	4 L Amber Glass Bottle 250 mL Glass Jar	4 °C 4 °C	7 1
8280A	Polychlorinated Dioxins and Furans by GC/MS	Water Soil/Other	4 L Amber Glass Bottle 250 mL Glass Jar	4 °C 4 °C	3 3
8318 pH 4 - 5	N-Methylcarbamate Pesticides by 7 Days HPLC	Water 40 Days Soil/Other	4 L Amber Glass Bottle 250 mL Glass Jar	4 °C; 0.1 N ClCH <sub>2</sub> CO <sub>2</sub> H, 4 °C	7
8330	Nitroaromatics and Nitramines by HPLC	Water Soil/Other	4 L Amber Glass Bottle 250 mL Glass Jar	4 °C 4 °C	7 1
TO-13A	PAHs in Filter Cartridges	PUF, Tenax, or XAD-2 Filter Cartridge		4 °C	7
TO-14A	VOC in Air	SUMMA <sup>®</sup> Canister			2
6321 (modified)	High Explosives by LC/MS/MS	Water Solid	Amber Glass/Teflon lined cap Amber Glass/Teflon lined cap	4 °C, 4 °C,	7 1

## GLOSSARY

### Attachment XX

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#### **Policy Statement for Sharing Laboratory-Performance Information Among DOE-AL Sites**

The DOE-AL Analytical Management Program (AMP), formerly the Field Sample Management Program (FSMP), is striving 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 DOE-AL 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 DOE-AL 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, DOE-AL facility personnel must bear in mind that the DOE's (and its subcontractor's) relationships with analytical laboratories are important partnerships. It is not in DOE'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 DOE-AL 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, IDL 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

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- 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 (e.g. EML and EMSL)
  - 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).

#### 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 perfectly 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 regarding any of the topics discussed above may be shared among projects under the system now being contemplated. If any project should decide to suspend a subcontract laboratory based on performance in any of these areas, other projects that also use that laboratory must decide how, if at all, the deficiency or deficiencies affect their samples.

#### Guidelines for Sharing Performance Information

With this in mind, the DOE-AL 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. This information should include any relevant quality assurance, quality control, technical,

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or reporting criteria. The laboratory being discussed should be copied on any such correspondence between DOE-AL facilities.

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- 2) Contact person(s) whose responsibility it is to transmit performance information to other DOE-AL 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) Whether the laboratory performance information received is applicable to the reviewer's project or not, the reviewer should attach a statement of applicability to the transmittal, with sufficient explanatory notes, before filing the transmittal.
- 5) 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.
- 6) Follow-up information regarding resolution of deficiencies should be transmitted to other DOE-AL facilities as quickly as possible so that the laboratory capacity originally in question can become available again.

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#### Policy Statement for Payment Reduction or Non-payment

In order to ensure that adequate value is obtained for dollars spent, it is sometimes necessary to impose a payment reduction or to elect non-payment 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 non-payment election are listed below.

- 1) If a laboratory fails to meet quality control criteria, and if reanalysis is precluded by expiration of holding times, then a payment reduction or non-payment 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 non-payment 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 non-payment 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 DOE-AL 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 non-payment 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 non-payment.

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- 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.

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- 8) Verified malfeasance, such as "dry-labing," should result in immediate suspension in addition to non-payment for the analyses in question.
- 9) Failure to submit periodic progress reports, IDL studies, performance evaluation data, or corrective action reports should not result in reduced payment for analysis data deliverables. Typically, laboratories are suspended pending compliance in such cases.